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PA-RPT-01034, R0	4/26/18	New	This report presents the results of the evaluation of cheesecloth alternatives exposed to nitric acid during ^{238}Pu aqueous processing and clean-up/decontamination activities

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1.0 INTRODUCTION

Heat source plutonium oxide (HS-PuO_2) enriched with 65% or greater plutonium-238, is recovered and purified using an aqueous nitric acid process. Historically, cheesecloth has been employed for small spill clean-up and for routine housekeeping. However, the 2016 Department of Energy (DOE) Operating Experience report, entitled Nitrate Waste Evaluations, stated that the mixing of nitrate wastes with organic absorbents, such as cheesecloth, may render transuranic waste unacceptable for disposal at WIPP in the future [1]. This report explores the feasibility of replacement of cheesecloth with an alternative sorbent wipe that has similar absorption and cleaning abilities and improved acid resistance.

Currently, cheesecloth is used for three different functions in HS-PuO_2 operations: spill cleanup; general housekeeping operations such as cleaning glovebox floors and windows, balances, furnaces, etc.; and decontamination of iridium cladded heat sources containing HS-PuO_2. Alternatives used for spill cleanup must be able to tolerate high neutron doses found in the HS-Pu gloveboxes. They must also be able to withstand exposure to nitric acid concentrations up to 15.8 M without decomposing. Wipes used for general housekeeping must also be durable enough to clean rough surfaces and remain intact when used for scrubbing. Wipes used for clad decontamination do not need to absorb large amounts of liquid but they must be able to withstand neutron exposures, temperatures of up to several hundred degrees Celsius, and exposure to acid solutions followed by mild scrubbing. Finally wipes used for any purpose must resist chemical reactions with nitric acid that could cause issues for waste disposal such as energetic exothermic reactions or flammable gas build-up.

The wipes selected for this study were ranked based on their usability (radiation tolerance, absorbency, integrity in nitric acid, and durability) as well as their chemical compatibility with nitric acid as demonstrated using several types of chemical analyses. Wipes were then ranked based on the combination of their usability and chemical compatibility scores.

1.1 Purpose

This report presents the results of the evaluation of cheesecloth alternatives exposed to nitric acid during ^{238}Pu aqueous processing and clean-up/decontamination activities. This study tested and compared the performance of cheesecloth and alternative materials under ^{238}Pu aqueous processing conditions to identify options that exhibited similar performance, increased radiation and thermal tolerance, and decreased reactivity with nitric acid. Based on testing performed by the Los Alamos National Laboratory Carlsbad Operations Difficult Waste Team, the four highest performing sorbent wipes were selected for further testing [2].Test results for each alternative were compared to cheesecloth and to the other alternatives to identify the best performers. The materials tested in this study are shown in Table 1.

1.1 Purpose (continued)

Table 1. Materials Tested

Product Name	Primary Chemical Composition	Manufacturer / Distributor	Product Part #
Cheese Cloth	Cellulose	Fisher	06-665-29
Kimtech Pure W4 Wipers	Polypropylene	Fisher	06-666-12
PBI Products	Sulfonated polybenimidazole	PBI Performance Products	M51015
Hazmat Sorbent SM Pad, Premium	Polypropylene	NPS Corporation	S2-70
Chamois	collagen	Acme Sponge and Chamois	TSX

1.2 Scope

The scope of this report is limited to the testing results for the four cheesecloth alternatives listed in Table 1 and cheesecloth. Initial testing of these alternatives included usability by determining absorbency, integrity in nitric acid, ^{238}Pu radiation tolerance, and durability. These test were used to down-select alternatives that were then chemically evaluated using elemental analysis- carbon, hydrogen nitrogen (CHN) analysis, differential scanning calorimetry / thermal gravimetric analysis (DSC/TGA), attenuated total reflection infra-red (ATR-IR) spectroscopy, and head space gas (HSG) analyses which included volatile organic compounds (VOC) off-gas and permanent gas analysis.

1.3 Background

In February 2014, a radiological release occurred at the WIPP site from a remediated nitrate salt waste drum from LANL. An Accident Investigation Board (AIB) concluded that an exothermic chemical reaction between organic materials and nitrate salts resulted in the release [3]. In June 2015, the Environment, Health, Safety, and Security (EHSS) Associate Undersecretary, Matthew Moury issued Operating Experience Level 2 (OE-2:2015-1), Evaluation of Nitrate-Bearing Transuranic Waste Streams, recommending the review of "all nitrate-bearing TRU waste streams that used neutralizers and/or absorbents for mitigation…" to determine if organics were used and to ensure the "ignitability characteristic of the remaining nitrate waste was mitigated." [4] In August 2016, clarification of the techniques to be used for evaluating hazards of nitrate wastes was provided in OE-3:2016-05, Nitrate Waste Evaluations.

1.3 Background (continued)

This document stated that future disposal at WIPP of mixtures of nitrate bearing TRU wastes with organic absorbents may not be allowed. In response, the Los Alamos National Laboratory Carlsbad Operations Difficult Waste Team performed a Sorbent Scoping Study [5], Oxidizer Scoping Study [6], and Testing of the Relative Oxidizing Hazard of Wipes and KMI Zeolite [2] to identify mixtures of oxidizers and sorbents that may be unsafe when mixed.

In addition, reactions of cheesecloth with materials and solutions containing ^{238}Pu have been implicated as a concern in several accidents at TA-55. In 1994, significant thermal decomposition and ignition of plutonium contaminated cheesecloth were reported as part of a Type C accident investigation [7]. In 2003, workers were exposed to ^{238}Pu from a release of airborne contamination from a degraded package that contained cellulose material and ^{238}Pu residues [8]. In 2016, evidence of thermal decomposition of several plastic materials temporarily in contact with cheesecloth used to absorb a spill of HS-PuO_2 dissolved in nitric acid was observed. [9]

Following the accidents in 1994 and 2003, numerous changes were implemented for the use of cheesecloth with ^{238}Pu. However no systematic study has been documented to identify other materials that are adequate alternatives to cheesecloth for ^{238}Pu operations that incorporate results identified in the LANL-CO Difficult Waste Team studies.

1.4 Summary of Findings

Kimtech wipes were found to be an excellent alternative to cheesecloth for low temperature applications (below 150°C). It exhibits similar usability to cheesecloth while showing little or no evidence of reactivity with nitric acid. Results were similar with the other polypropylene wipe, Hazmat pads, with the exception of an unexplained volatile organic off-gas that was observed in small quantities. No evidence of reaction with nitric acid was detected for either the Kimtech wipes or Hazmat pads. PBI was identified as the best alternative for temperatures above 150°C. However, PBI had poor absorption performance in comparison to cheesecloth and the polypropylene alternatives. The infrared (IR) spectrum of PBI exposed to nitric acid showed evidence of changes that are indicative of interstitial solvated nitric acid not nitration, although this is not definitive. Chamois reacted with nitric acid at most concentrations tested and is not a viable alternative.

2.0 ACRONYMS AND DEFINITIONS

2.1 Acronyms

Term	Definition
A_f	Absorbency Factor
ATR-IR	Attenuated Total Reflectance Infrared Spectroscopy
CHN	Carbon, Hydrogen, Nitrogen (Analysis)
DOE	Department of Energy
DSC	Differential Scanning Calorimetry
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatography
HF	Hydrofluoric Acid
HNO_3	Nitric Acid
HSG	Head Space Gas
IWD	Integrated Work Document
µg	Microgram
m/z	Mass-to-charge ratio
mm	Millimeter
MS	Mass Spectroscopy
^{238}Pu	Plutonium-238
TCD	Thermal Conductivity Detector
TGA	Thermogravimetric Analysis
VOC	Volatile Organic Compounds

2.2 Definitions

Term	Definition
Absorbency	Quality of a material to absorb liquids.
Cellulose	Term used to describe carbon- and hydrogen- containing products, such as cheesecloth.
Clad	An iridium-tungsten container comprised of two cylindrical cups, welded together, used to encapsulate the Pu-238 fuel pellet.
Enthalpy	The sum of the internal energy of s system (substance) plus the product of the pressure and volume.
Functional Groups	A group of reactive atoms that define the characteristics of a compound.
Head Space Gas	The gases present above vapor-emitting liquids or solids in a closed container.
Heat Capacity	The amount of heat it takes to raise the temperature of a compound by one degree C.
Nitrate Esterification	A reaction in which a nitrate ester ($RONO_2$, where R represents an organic constituent) and water are formed typically from the reaction of nitric acid and an alcohol.
Permanent Gases	Nitrogen, oxygen, carbon dioxide, and carbon monoxide.
Z Number	The atomic number of a chemical element.

3.0 RESULTS

All alternatives were evaluated for usability with multiple techniques as described below. To reduce the number of tests to be performed, samples were screened for their absorbency and integrity to withstand reaction when exposed to nitric acid. Samples passing these screens were evaluated for the remaining usability criteria and were then chemically evaluated.

3.1 Screening Results

Four alternatives were qualitatively evaluated for usability as compared to cheesecloth using these criteria:

1) ^{238}Pu radiation tolerance
2) durability
3) absorbency
4) integrity in nitric acid

Alternatives were scored using the criteria in Table 2.

Table 2. Scale for Usability Scoring

Score	1	2	3	4	5
Rad Tolerance	Decomposed/ Charred after 3 days	Decomposed/ Charred after 2 weeks	Stiffness, brittleness, or flaking after 4 weeks	Yellowing only after 4 weeks	No change after 4 weeks
Durability	Tears on initial wiping	Cannot wring out material	Tears during use after rinsing/ wringing	Less effective after rinsing/ wringing	No observed change
Absorbency (A_f)	<2	≥2	≥4	≥ 6	≥ 8
Integrity	Decomposition	Charring/ Shriveling	Stiffness	Yellowing	No observed changes

3.2 ^{238}Pu Radiation Tolerance

The neutron radiation tolerance of the four alternatives were evaluated by placing the sample in the dropbox adjacent to the ^{238}Pu glovebox for 28 days. Although ^{238}Pu is an alpha emitter, the alpha particles it generates can also undergo α,n reactions with low z number materials, especially fluorine. Hydrofluoric acid is used to catalyze the digestion of HS-PuO_2 with nitric acid which results is a high neutron emission rates during aqueous processing. Therefore, materials placed in the ^{238}Pu aqueous glovebox can be exposed to both alpha and neutron radiation but because there was several feet between the reaction vessels in the glovebox and the dropbox, samples in the dropbox were likely to be exposed the neutron radiation only.

The samples were observed in the dropbox over a period of 41 days. An electronic personal dosimeter was placed alongside the samples to record the cumulative radiation dose the sample received. The batteries died on day 42 and due to lack of room availability, they were not able to be replaced immediately. The cumulative dose the samples received after 41 days was 9000 mrem. Photographs of samples were taken on day 5 and on day 29 which showed no observable change. Visual observation on day 41 showed no changes as well.

Although, operators had previously reported flaking and tackiness occurring with polypropylene wipes such as Hazmat and Kimtech, no changes were observed with these alternatives or any of the other alternatives over the period of observation. Therefore all alternatives received the highest ranking of 5 in Table 3 using the criteria in Table 4. The lack of evidence of change of the alternatives suggests that all materials are resistant, at least in the short term, to radiolytic degradation by neutron radiation. As mentioned above, it is unlikely that the samples were exposed to alpha radiation, therefore it is not possible to rule out that the previously reported degradation resulted from alpha radiation exposure. Also, due to the 238Pu contamination within the dropbox, it was not possible to perform chemical analysis on these samples.

Table 3. Radiation Tolerance Scoring

Alternative	**Cheese Cloth**	**Kimtech**	**PBI**	**Hazmat**	**Chamois**
Rad Tolerance Score	5	5	5	5	5

Table 4. Scale for Radiation Tolerance Scoring

Score	**1**	**2**	**3**	**4**	**5**
Rad Tolerance	Decomposed/ Charred after 3 days	Decomposed/ Charred after 2 weeks	Stiffness, brittleness, or flaking after 4 weeks	Yellowing only after 4 weeks	No change after 4 weeks

3.3 Durability

Cheesecloth and four alternatives were evaluated for durability using a simulated spill of sodium chloride on the floor of an uncontaminated, demonstration only glovebox. The simulated spills were prepared by pouring the salt into a short pile of roughly 4 inches in diameter and then adding water until the salt formed a thick paste. The paste was allowed to dry at least 24 hours to form a hard cake.

Alternatives that were purchased in sizes larger than a piece of cheesecloth (i.e. Hazmat Pads and PBI) were cut to approximately the same size as a piece of cheesecloth, nominally 12 inch squares. The chamois and Kimtech samples were used as obtained from the vendor. Each sample was dipped into a large beaker of water until saturated, wrung out using glovebox gloves, and then used to wipe up approximately one third of a salt cake. As the samples became covered in salt or if the salt began to smear across the glovebox, the wipes were rinsed and wrung out before continued use. Observations about absorption, ease of wringing, ease of wiping up the crusted salt, and ease of picking up dispersed salt particles dislodged from the spill during wiping were observed. This process was repeated by a second operator who made independent observations that were found to be consistent with the observations of the first operator.

In addition, following the glovebox experiments, the samples were removed from the glove box and rinsed. Each sample was used to wipe down benchtops around the sink area to provide additional observations about use for clean-up.

All of the alternatives were found to be durable enough to wipe up the abrasive salt spills. They were all wrung out multiple times without tearing. Upon exposure to water, the chamois sample became slimy and left the rinse water brown. This is believed to be from residual fish oil from its curing process. It was also observed that the bottom layer of the Hazmat was found to catch on countertop edges which pulled it apart from the top layer, making it more difficult to use. Based on these observations, durability scores were determined for each alternative as shown in Table 5, using the criteria from Table 6.

Table 5. Durability Scoring

Alternative	**Cheese Cloth**	**Kimtech**	**PBI**	**Hazmat**	**Chamois**
Durability Score	5	5	5	4	4

Table 6. Scale for Durability Scoring

Score	**1**	**2**	**3**	**4**	**5**
Durability	Tears on initial wiping	Cannot wring out material	Tears during use after rinsing/ wringing	Less effective after rinsing/ wringing	No observed change

3.4 Absorbency

To determine absorbency, 4 in^2, samples of each alternative plus cheesecloth were weighed, saturated with 1M nitric acid, and reweighed. This was repeated using 5M, 10M, and 15.8M nitric acid. The absorbency factors, shown in Figure 1, were determined as a measure of absorbency using the equation:

$$A_f = \text{wet sample mass / dry sample mass.}$$

Absorbency scoring is provided in Table 7 along with the scale in Table 8.

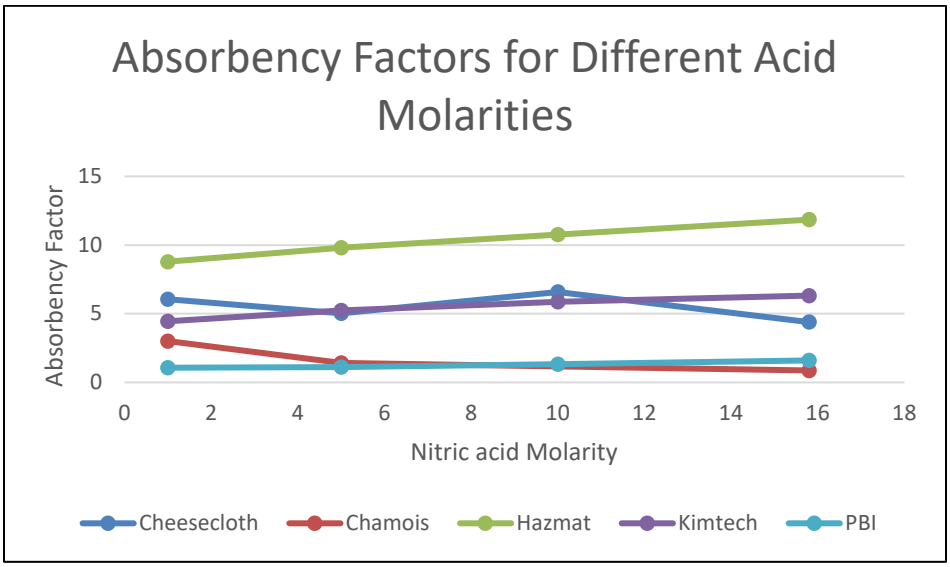


Figure 1. Absorbency factors for alternatives exposed to 1M, 5M, 10M, and 15.8M nitric acid.

As seen in Figure 1, Hazmat showed higher absorbency than cheesecloth while Kimtech absorbency was very similar to cheesecloth. Chamois absorbency was roughly half of cheesecloth using 1M nitric acid and decreased with increasing acid molarity. PBI showed almost no absorbency at 1M nitric acid and showed only a small increase with increasing molarity. The wet to dry ratio for chamois with 10M nitric acid is not reported because of a sampling error. Scoring of the alternatives and cheesecloth at each concentration is shown in Table 7.

Table 7. Absorbency Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat	Chamois
1M HNO3 Absorbency	4	3	1	5	2
5M HNO3 Absorbency	3	3	1	5	1
10M HNO3 Absorbency	4	3	1	5	1*
15.8M HNO3 Absorbency	3	4	1	5	1

* Score for chamois with 10M nitric acid is extrapolated because of a sampling error

Table 8. Scale for Usability Scoring

Score	1	2	3	4	5
Absorbency (A_f)	<2	≥2	≥4	≥ 6	≥ 8

3.5 Integrity in Nitric Acid

The samples assessed for absorbency screening were monitored to evaluate their integrity in nitric acid. Samples were assessed after 1 day, 2 days, and 25 days. Photos of samples after 2 days and at 25 days are shown in Appendix 1.

Samples of Hazmat, Kimtech, and PBI remained unchanged at all acid molarities for the duration of the observation period, as indicated by the maximum scores shown in Table 5. The cheesecloth sample exposed to 1M nitric acid remained unchanged during the observation period. Cheesecloth samples exposed to 5M acid and above began showing slight discoloration as the samples dried. This discoloration was more pronounced at higher molarities, as seen in Figure 2. Also, the cheesecloth exposed to 10M and 15.8M acid concentrations began to stiffen as they dried and the 15.8M sample showed flaking at the end of the observation period.

Table 9. Integrity in Nitric Acid Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat	Chamois
1M HNO3 Integrity	4	5	5	5	2
5M HNO3 Integrity	4	5	5	5	2
10M HNO3 Integrity	4	5	5	5	1
15.8M HNO3 Integrity	1	5	5	5	1

Table 10. Scale for Usability Scoring

Score	**1**	**2**	**3**	**4**	**5**
Integrity	Decomposition	Charring/ Shriveling	Stiffness	Yellowing	No observed changes

3.5 **Integrity in Nitric Acid** (continued)

Figure 2. Cheesecloth exposed to 15.8M nitric acid for 25 days.

The chamois samples performed very poorly upon exposure to all molarities of nitric acid. At all molarities, the samples shriveled upon contact. At molarities of 10M and higher, the chamois melted after 1 day of exposure, as shown in Figure 3.

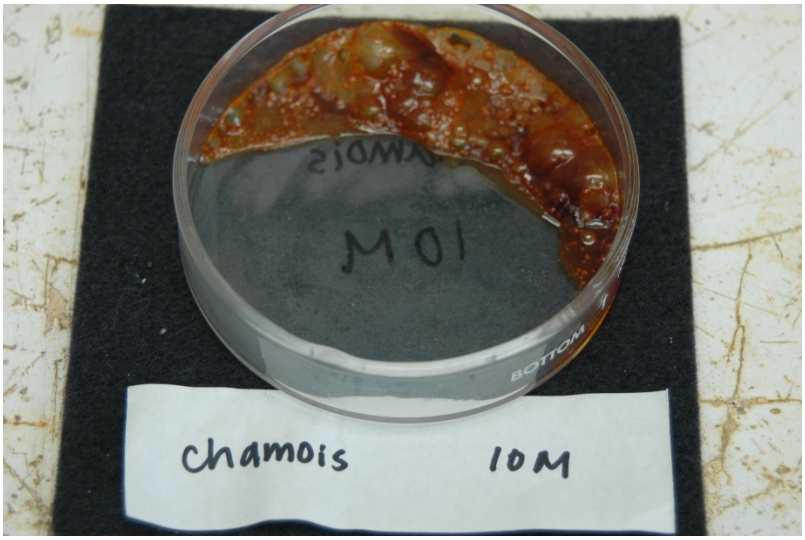


Figure 3. Chamois exposed to 10M nitric acid for 2 days.

3.6 Screening Results

Based on the results of this testing, show in Table 11, chamois was excluded from further testing. Although PBI scored poorly for absorbency, it was retained for further testing to determine its suitability for clad decontamination which is less dependent on absorbency.

Table 11. Usability Subtotals and Usability Screening Scores

Alternative	**Cheese Cloth**	**Kimtech**	**PBI**	**Hazmat**	**Chamois**
1M HNO3 Subtotal	18	18	16	19	13
1M HNO3 Usability Score	4.5	4.5	4	4.75	3.25
5M HNO3 Subtotal	17	18	16	19	12
5M HNO3 Usability Score	4.25	4.5	4	4.75	3
10M HNO3 Subtotal	18	18	16	19	11
10M HNO3 Usability Score	4.5	4.5	4	4.75	2.75
15.8M HNO3 Subtotal	14	19	16	19	11
15.8M HNO3 Usability Score	3.5	4.75	4	4.75	2.75

4.0 CHEMICAL ANALYSES

Based on the screening results, Hazmat, Kimtech, and PBI advanced for additional analyses. Samples were analyzed for head space gases by volatile organic compounds (VOCs) off-gas analysis and permanent gases (nitrogen, oxygen, carbon dioxide, and carbon monoxide) analysis. Head space gas analysis is useful for identifying volatile gases formed from the reaction of cheesecloth or alternatives with nitric acid. In particular, a build-up of flammable gases such as hydrogen or low molecular weight VOCs, such as methane, upon reaction of an alternative with nitric acid would raise disposal concerns about the alternative.

Carbon, hydrogen, and nitrogen (C,H,N) elemental analysis was selected to show if significant amounts of nitric acid remain in contact or react with alternatives. Fourier transform infrared spectroscopy (FT-IR) is a useful technique for identifying the presence of functional groups in organic compounds and the use of attenuated total reflection (ATR) allows this capability to be extended to solid samples such as wipes. Therefore this technique was selected to identify if an alternative reacted with nitric acid.

Differential scanning calorimetry (DSC) was used to measure how the heat capacity of a material changed with temperature. Thermal gravimetric analysis (TGA) measures mass changes of a sample as a function of temperature. Combined, these two techniques can provide information about thermal characteristics of a material such as phase changes, thermal transitions, heat capacities, desorption and decomposition.

Alternatives were scored using the criteria in Table 12. Results of these analyses are presented below and descriptions of the analysis techniques are presented in Appendix 3.

Table 12. Scale for Chemical Analysis Scoring

Score	1	2	3
Head Space Gas VOC	Evidence of flammable gases by GC/TCD and/or GC/MS analysis	Evidence of non-flammable gases or volatile organic compounds by GC/TCD and/or GC/MS analysis	Same as untreated sample
Permanent Gas	Major Evidence of reactions (>20% difference from air concentrations)	Minor Evidence of reactions (10-20% difference from air concentrations)	Not change compared to Air
CHN	Elevated N values after washing indicating nitration	Changes in CHN ratios indicating decomposition	Washed sample same as untreated sample
DSC/TGA	Reduction of decomposition temperature by 50 ℃ or more	Evidence of desorption of HNO_3 and/or H_2O	Same as untreated sample
FTIR-ATR	Evidence of nitrate absorption bands indicating nitration	Evidence of structural changes indicating decomposition, crosslinking, etc.	Same as untreated sample

4.1 Volatile Organic Compounds (VOC) Off-gas Analysis

Samples of the untreated and 15.8M HNO_3 treated cloths were analyzed in triplicate for volatile organic compounds (VOC) off-gas products using static headspace extraction with gas chromatography coupled to quadrupole mass spectrometry (HS-GC-qMS). The purpose of this testing was to identify flammability concerns due to the presence of volatile oxidation products formed upon exposure to nitric acid.

No detectable chromatographic peaks were found for the analyses of the untreated cheesecloth or the three alternatives. For all of the samples treated with nitric acid, there were two abundant peaks around 3.6 minutes. These peaks are attributed to nitrogen dioxide (NO_2) which was produced by the thermal decomposition of HNO_3 and from HNO_3 that was transferred to the GC and then converted to NO_2 during the temperature ramp. For the Hazmat pads, there was also a peak at 9.9 minutes for all three Hazmat sample runs. Mass spectrometry software tentatively identified this substance as 3,3-diethyl-pentane by comparison with the National Institute of Standards and Technology (NIST) mass spectral library. Based on discussions with the manufacturer, 3,3-diethyl-pentane or similar substituted hydrocarbons are not typical byproducts of the manufacturing process for Hazmat pads. The pigment, zinc ferrite, and the surfactant, sodium bis(2-ethylhexyl) sulfosuccinate were the only low levels contaminants reported by the manufacturer.

The average integrated peak abundances of the analyses for each sample type are shown in Table 13. No other peaks of significant abundance were identified. These results are consistent with the absorbency findings using concentrated nitric acid that indicated that the HAZMAT and Kimtech wipes could absorb more acid than cheesecloth while the PBI wipes had poor absorption ability. The lack of any other significant VOC gases for cheesecloth, PBI, and Kimtech wipes indicates that little if any volatile oxidation products were formed upon reaction of cheesecloth or the alternatives with nitric acid. It is unclear whether the peak at 9.9 minutes for Hazmat is an oxidation product or a low level contaminant in the wipe.

Table 13. VOC Off-Gas Peak Abundance Results

Sample	HS Peak 3.6 min	HS Peak 9.9 min
Cheesecloth	8,319,047	0
PBI	3,870,770	0
Hazmat	12,011,351	81,206
Kimtech	11,818,405	0

Based on the results above, the alternatives were ranked as shown in Table 14 using the Table 15 criteria.

Table 14. Volatile Organic Compounds (VOC) Off-gas Analysis Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat
Score	3	3	3	2

4.1 Volatile Organic Compounds (VOC) Off-gas Analysis (continued)

Table 15. Scale for Quantitative Scoring

Score	1	2	3
Criteria	Evidence of flammable gases by GC/TCD and/or GC/MS analysis	Evidence of non-flammable gases or volatile organic compounds by GC/TCD and/or GC/MS analysis	Same as untreated sample

4.2 Permanent Gas Analysis

Following VOC analysis, headspace samples were analyzed by gas chromatography for the permanent gases: hydrogen, helium, oxygen, nitrogen, nitrous oxide, carbon dioxide, and nitric oxide carbon monoxide. Samples of both untreated and 15.8M HNO_3 treated cheesecloth and alternatives were analyzed. After dilution correction for the carrier gas from the VOC analysis, a small amount of carbon monoxide was found to have been produced in all the samples treated with nitric acid. This is consistent with a small amount of oxidation of the wipe by nitric acid. No other statistically significant changes in permanent head space gas composition were detected between the untreated and treated samples. Table 3 show the average percent increase in carbon monoxide for the treated samples.

Table 16. Average Percent Carbon Monoxide Increase of Treated versus Untreated Samples using GC Permanent Gas Analysis

Sample	Difference in Treated vs Untreated Samples
Cheesecloth	0.18%
PBI	0.20%
Hazmat	0.17%
Kimtech	0.18%

Table 17. Permanent Gas Analysis Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat
Score	3	3	3	3

Table 18. Scale for Quantitative Scoring

Score	1	2	3
Criteria	Major Evidence of reactions (>20% difference from air concentrations)	Minor Evidence of reactions (10-20% difference from air concentrations)	No change compared to air

4.3 Carbon Hydrogen Nitrogen (CHN) Elemental Analysis

Samples of the untreated and 15.8M HNO_3 treated cloths were analyzed for carbon, hydrogen, and nitrogen elemental composition using a Perkins Elmer 2400 Series II Elemental Analyzer configured in the CHN mode. The purpose of these analyses was to identify changes in nitrogen content in samples treated with concentrated nitric acid versus untreated samples. An increase in nitrogen values would be expected if nitration of the samples occurred. The cheesecloth showed an increase in the nitrogen values indicating a small amount of nitrate esterification (i.e. reaction of alcohol on cellulose with a nitrate group) or the presence of residual nitric acid. The PBI also showed an increase in nitrogen content. This is believe to result from residual nitric acid and is discussed further in the infrared spectroscopy section. The small amount of nitrogen found in the Hazmat and Kimtech samples are believed to arise from residual nitric acid in the sample after drying at 60 °C.

Table 19. Average Percent Nitrogen Increase of Treated versus Untreated Samples using C,H,N Analysis

Sample	Difference in Treated vs Untreated Samples
CC	1.17
HZMT	0.21
KMTC	0.73
PBI	3.58

Table 20. C,H,N Elemental Analysis Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat
Score	1	3	1	3

Table 21. Scale for Quantitative Scoring

Score	1	2	3
Criteria	Elevated N values after washing indicating nitration	Changes in CHN ratios indicating decomposition	Washed sample same as untreated sample

5.0 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ATTENUATED TOTAL REFLECTION (ATR)

FT-IR ATR was performed on both untreated cheesecloth and alternatives and cheesecloth and alternatives treated with 15.8M nitric acid.

Cheesecloth is composed primarily of cellulose which has the structure shown below:

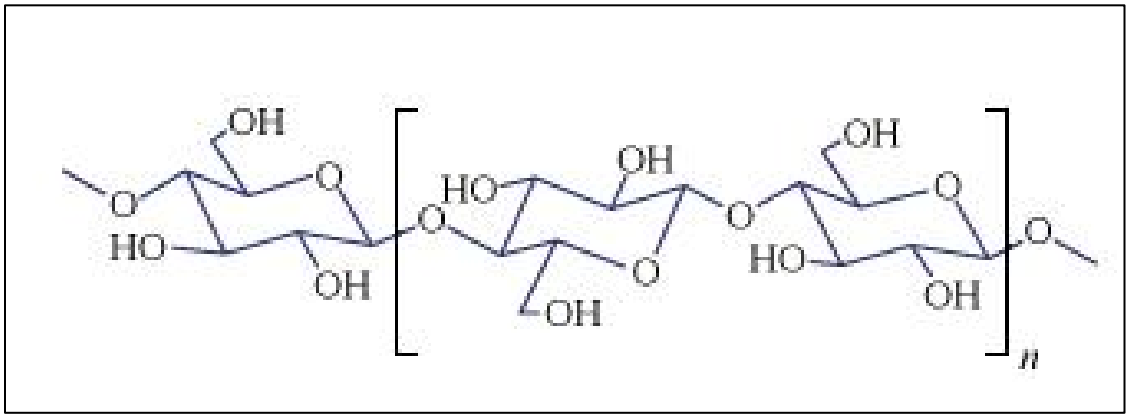


Figure 4. Chemical Composition of Cellulose [10]

Cheesecloth showed evidence for the formation of new functional groups in the IR spectrum of the treated material. New peaks at 1734 cm^{-1}, 1637 cm^{-1}, 1281 cm^{-1}, 853 cm^{-1} are found in the spectrum of cheesecloth treated with concentrated nitric acid that are not present in the untreated sample spectrum. Saunders and Taylor reported that the region from 1800 to 1600 cm^{-1} is valuable for the identification of the nitrate functional group (O-N-O) in cellulose nitrate because absorption of the antisymmetric stretching mode occurs in this region [11]. Identification of the peak at 1734 cm^{-1} is confounded by the reports identifying peaks in this region as C=O stretching vibrations. C=O bonds might be detectable if nitric acid were oxidizing the cheesecloth [12, 13]. Numerous studies support the identification of the peak at 1637 cm^{-1} as belonging to the nitrate group [11, 13-16]. However several other reports identify peaks in this region as OH bends [12, 15]. The peaks at 1281cm^{-1} and 853cm^{-1} have been identified as characteristic nitrate absorption bands [16, 17]. These spectral changes indicate that nitration and/or oxidation of the cheesecloth occurred.

5.0 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ATTENUATED TOTAL REFLECTION (ATR) (continued)

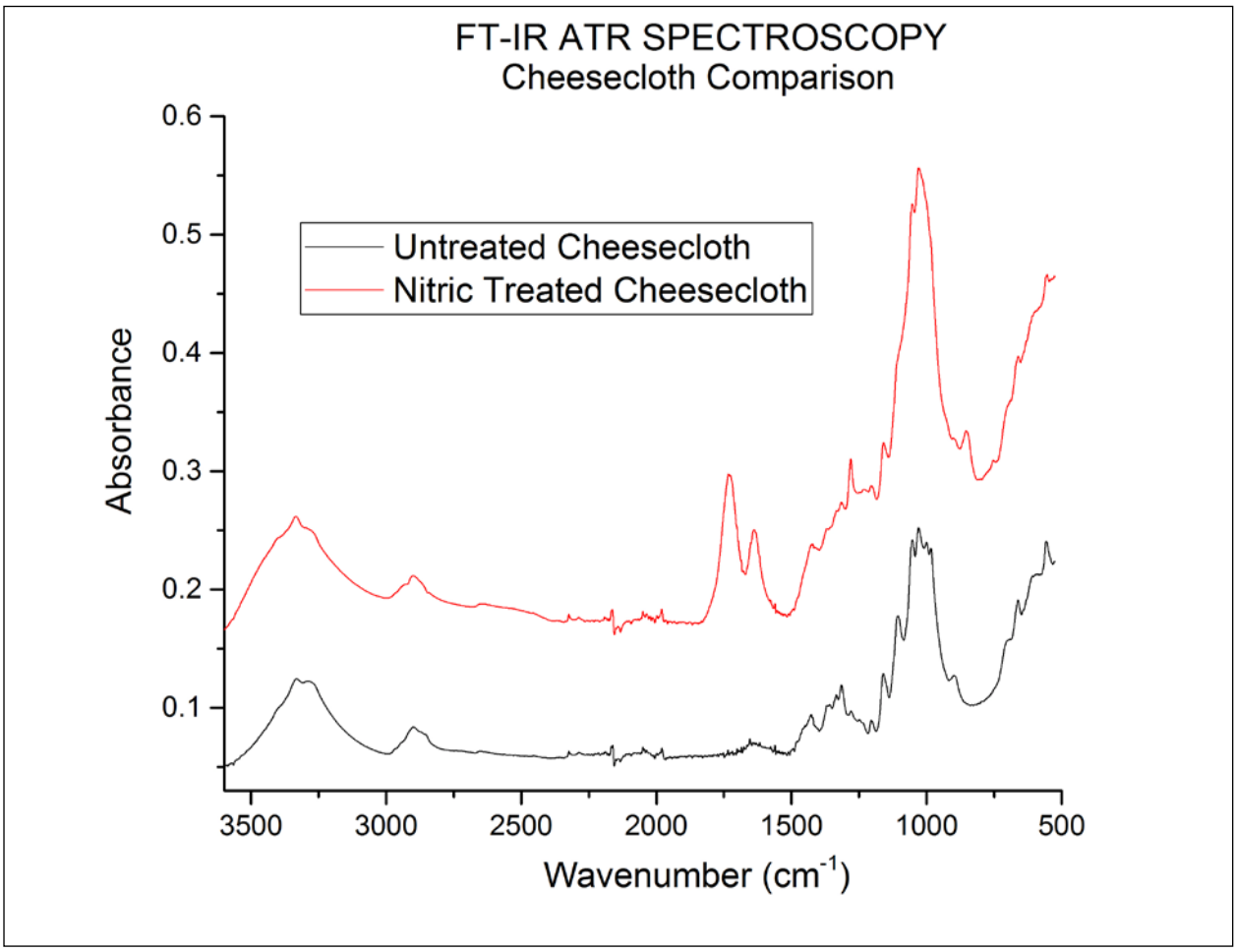


Figure 5. FT-IR of 15.8M HNO_3 Treated Cheesecloth and Untreated Cheesecloth

Hazmat pads and Kimtech wipes are both composed of polypropylene, with the composition shown in Figure 6.

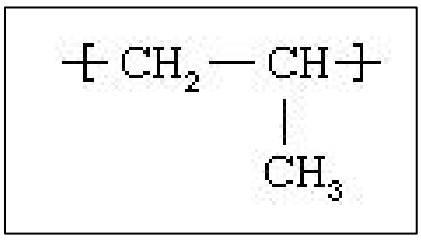


Figure 6. Chemical Composition of Polypropylene [18]

5.0 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ATTENUATED TOTAL REFLECTION (ATR) (continued)

The IR spectra of the polypropylene wipes, showed no changes between the nitric acid treated and untreated samples. No evidence of the contaminant or possible oxidation product, 3,3-diethylpentane that was detected in the head space gas analysis was identified in the IR spectrum of the Hazmat pads. However the IR spectrum 3,3-diethylpentane shows absorption bands in the same region as polypropylene so identification of this material using IR would not be expected. [19]

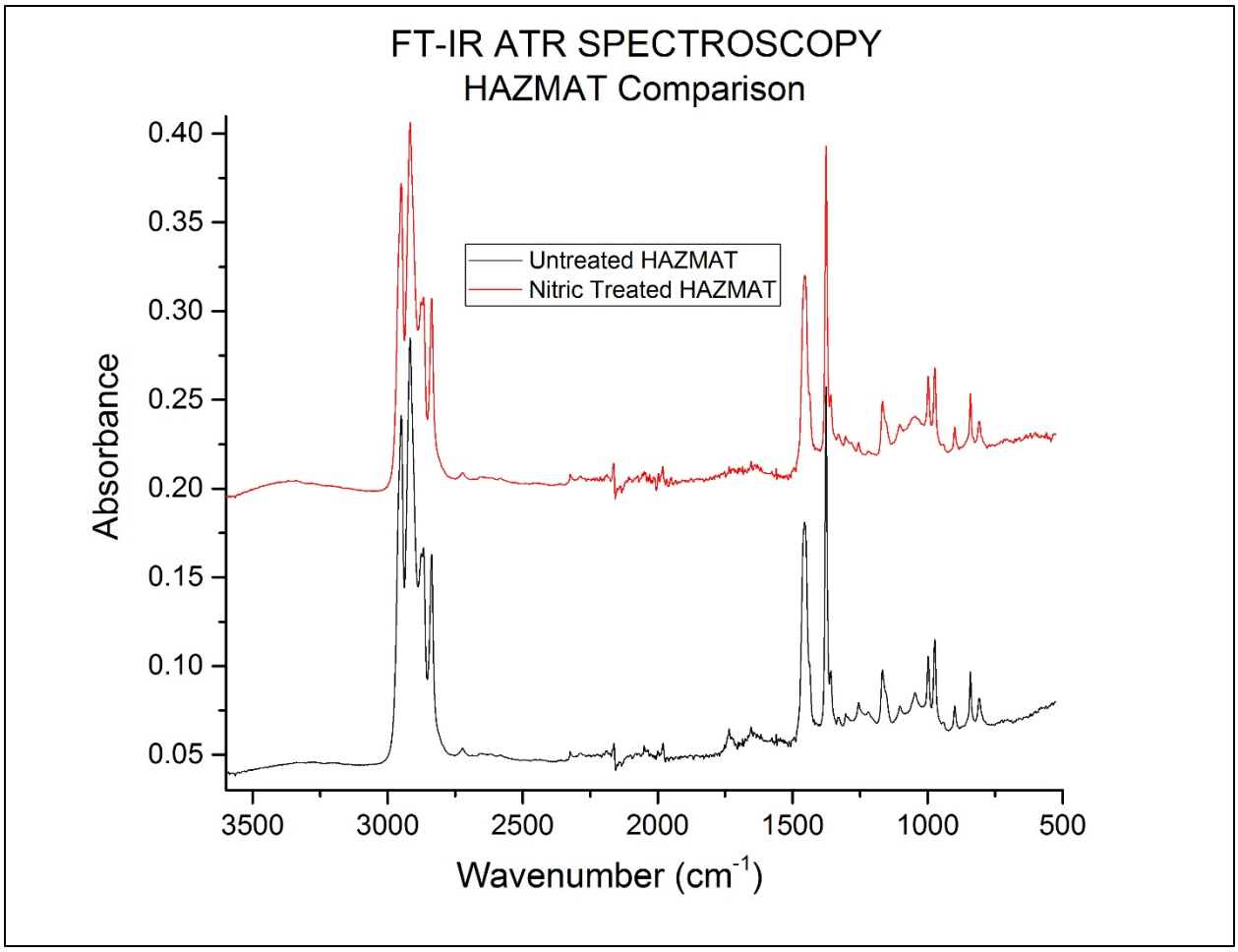


Figure 7. FT-IR of 15.8M HNO_3 Treated and Untreated HAZMAT Wipes

5.0 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ATTENUATED TOTAL REFLECTION (ATR) (continued)

Figure 8. FT-IR of 15.8M HNO_3 Treated and Untreated Kimtech Wipes

PBI is reported to be a sulfonated polybenzimidazole. The exact structure of the material is proprietary so the structure of polybenzimidazole without sulfate functional groups is shown in Figure 9.

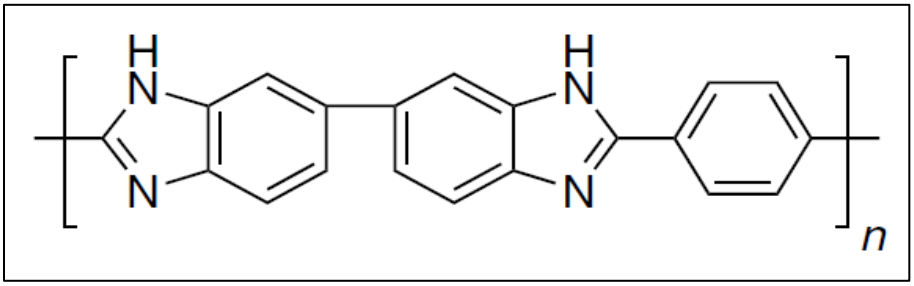


Figure 9 Chemical Composition of unsulfonated PBI [20]

The interpretation of the IR spectra of the untreated PBI is complicated due to the presence of many different types of functional groups (aromatic rings, imidazole, sulfonate, etc.) with absorbances in overlapping regions. A qualitative comparison of the untreated sample with the treated sample shows several important changes.

For the nitric acid treated PBI sample, two new absorbances are seen from 1400-1300 cm^{-1}. A peak at 1629 cm^{-1} is observed in the treated sample that has a greater intensity then the peak at 1625 cm^{-1} in the untreated sample. Goebbert and coworkers studied the IR spectra of hydrated nitrate ions ($NO_3^-(H_2O)_n$ clusters, n=1-6) and reported NO_3^- antisymmetric stretching of centered around 1350 cm^{-1} [21]. This stretching mode was observed as either a doublet or a singlet depending on the number of water molecules in the cluster similar to those seen for the two new peaks in spectrum for the treated PBI. In addition a weak band in the mid 1600 cm^{-1} region was identified in the Goebbert report that was assigned to the water bending modes similar to the peak seen at 1629 cm^{-1}.

Furthermore, nitro-substituted polybenzimidazole has been reported by Choi and coworkers from the reaction of PBI (non-sulfonated) with a nitric/sulfuric acid mixture [22]. This substance was found to have strong absorbances at 1517 cm^{-1} and 1332 cm^{-1} due to nitration of the aromatic ring that are not seen in the sulfonated PBI sample treated with nitric acid. The difference between the Choi results and the treated PBI spectrum, along with the similarities of the treated PBI spectra with hydrated nitrate ion spectra reported by Goebbert, suggest that the sulfonated PBI contains interstitial dissociated nitric acid or possibly weak hydrogen bonding between PBI and dissociated nitric acid.

5.0 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ATTENUATED TOTAL REFLECTION (ATR) (continued)

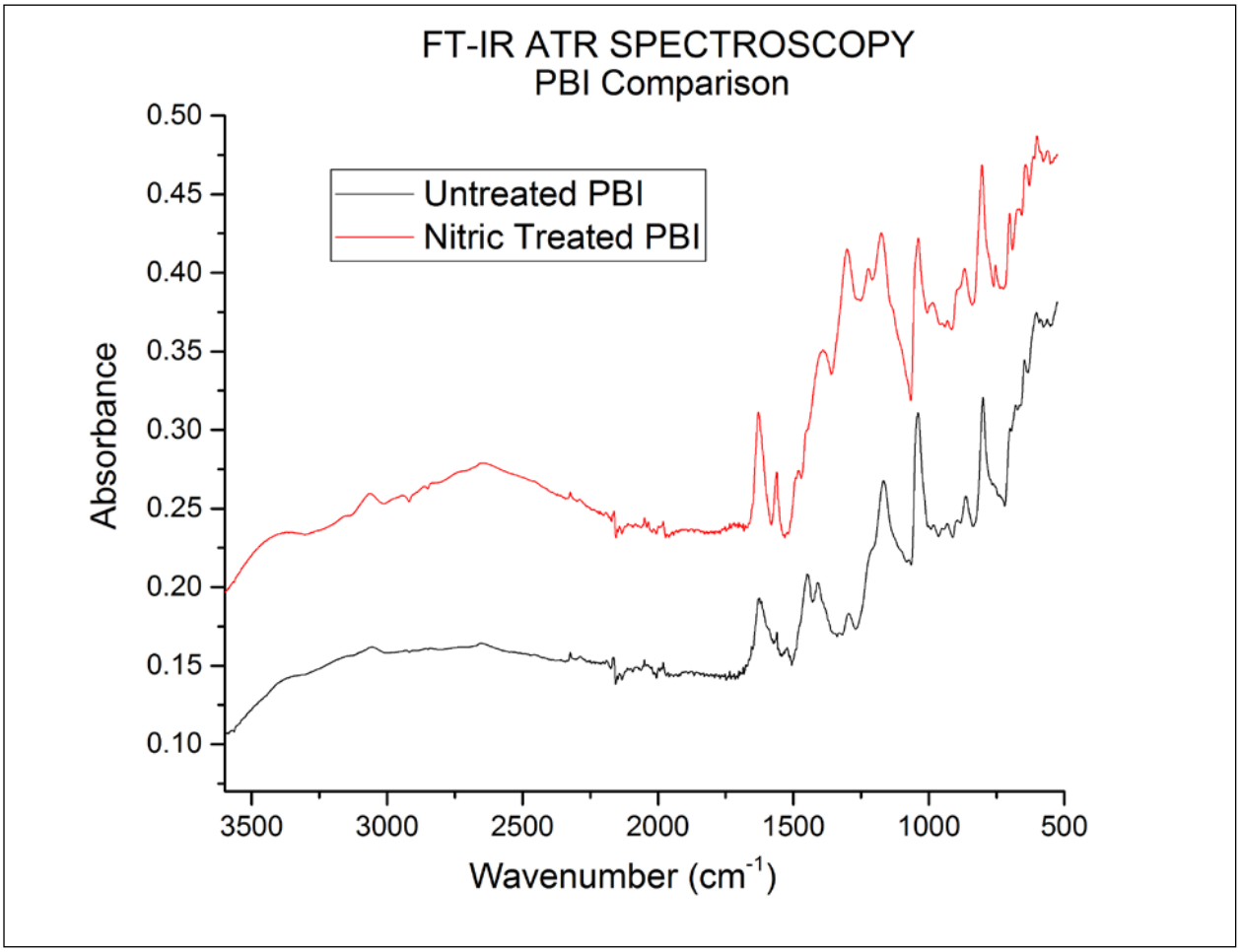


Figure 10. FT-IR of 15.8M HNO_3 Treated and Untreated PBI

Table 22. FT-IR ATR Scoring

Alternative	**Cheese Cloth**	**Kimtech**	**PBI**	**Hazmat**
Score	1	3	2	3

Table 23. Scale for Quantitative Scoring

Score	**1**	**2**	**3**
Criteria	Evidence of nitrate absorption bands indicating nitration	Evidence of structural changes indicating decomposition, crosslinking, etc.	Same as untreated sample

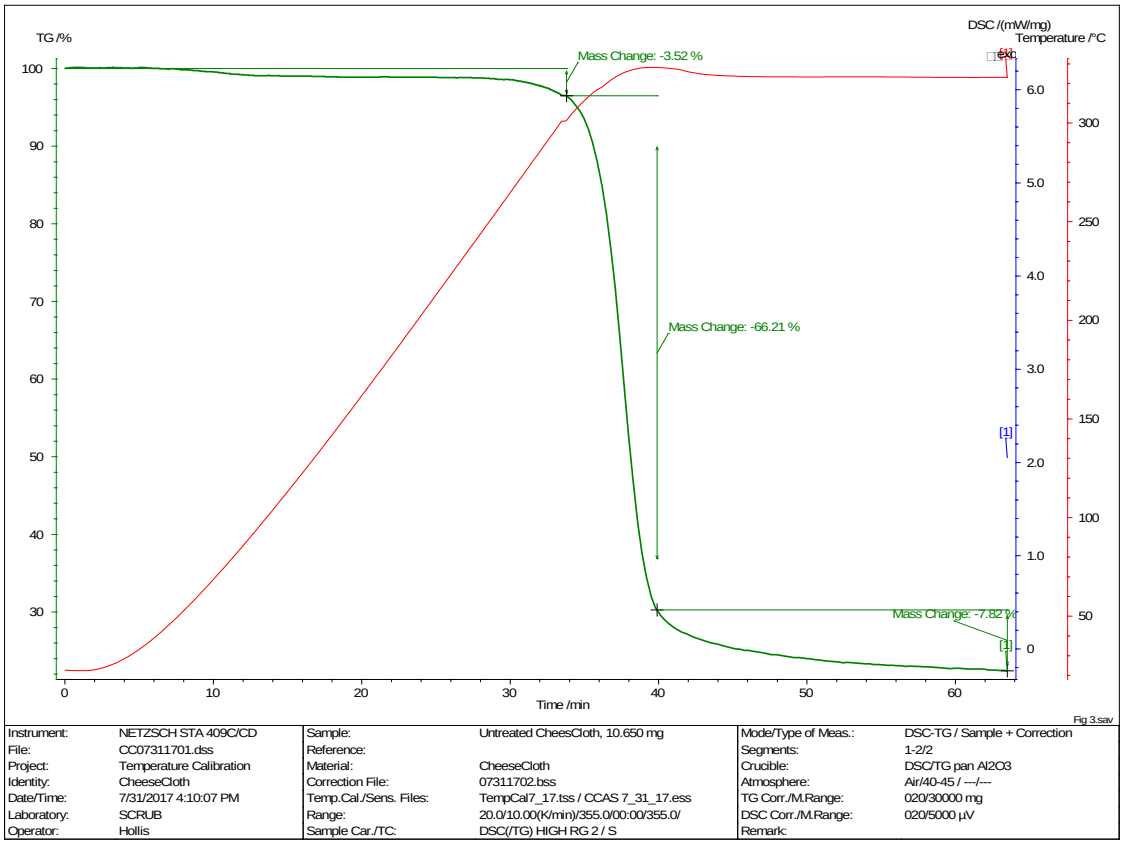
6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC)

To understand changes that occur with increased temperature, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed on samples of the untreated and HNO_3 treated materials using a Netzsch TGA/DSC Model 410 equipped with a Skimmer mass spectrometer system for produced gas identification..

Two experimental temperature profiles were used for the TGA/DSC analysis. First, the sample was analyzed from 30°C to 300°C at a 10°C /min ramp rate. The sample was then held at 300°C for 30 minutes. This was done to simulate waste packaging conditions and temperature maximums referenced in CALC: C-CDE-17-001.[23] The second profile was similar with a 30°C starting temperature, a 10°C /min ramp rate but a final temperature of 600°C with no hold time. This profile was used in order to apply DSC enthalpy calibration data that cannot be applied if a temperature hold is employed.

Results from the 300°C hold profile for cheese cloth (CC) showed similar results reported in CALC: C-CDE-17-001.[23] The untreated CC, (Figure 11) showed an initial thermal oxidation which ended at approximately 300°C, with a mass loss of 3.52% based on review of the DSC curve seen in the appendix.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)



Instrument:	NETZSCH STA 409C/CD	Sample:	Untreated CheesCloth, 10.650 mg	Mode/Type of Meas.:	DSC-TG / Sample + Correction
File:	CC07311701.dss	Reference:		Segments:	1-2/2
Project:	Temperature Calibration	Material:	CheeseCloth	Crucible:	DSC/TG pan Al2O3
Identity:	CheeseCloth	Correction File:	07311702.bss	Atmosphere:	Air/40-45 / ---/---
Date/Time:	7/31/2017 4:10:07 PM	Temp.Cal./Sens. Files:	TempCal7_17.tss / CCAS 7_31_17.ess	TG Corr./M.Range:	020/30000 mg
Laboratory:	SCRUB	Range:	20.0/10.00(K/min)/355.0/00:00/355.0/	DSC Corr./M.Range:	020/5000 µV
Operator:	Hollis	Sample Car./TC:	DSC(/TG) HIGH RG 2 / S	Remark:	

Figure 11. TGA of Untreated Cheesecloth (profile 1)

Continued thermal oxidation between 300°C and 328°C accounted for the primary mass loss and resulted in an additional 66.21% loss. A final thermal decomposition of the material continued over the hold temperature resulting in 7.28% mass loss.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

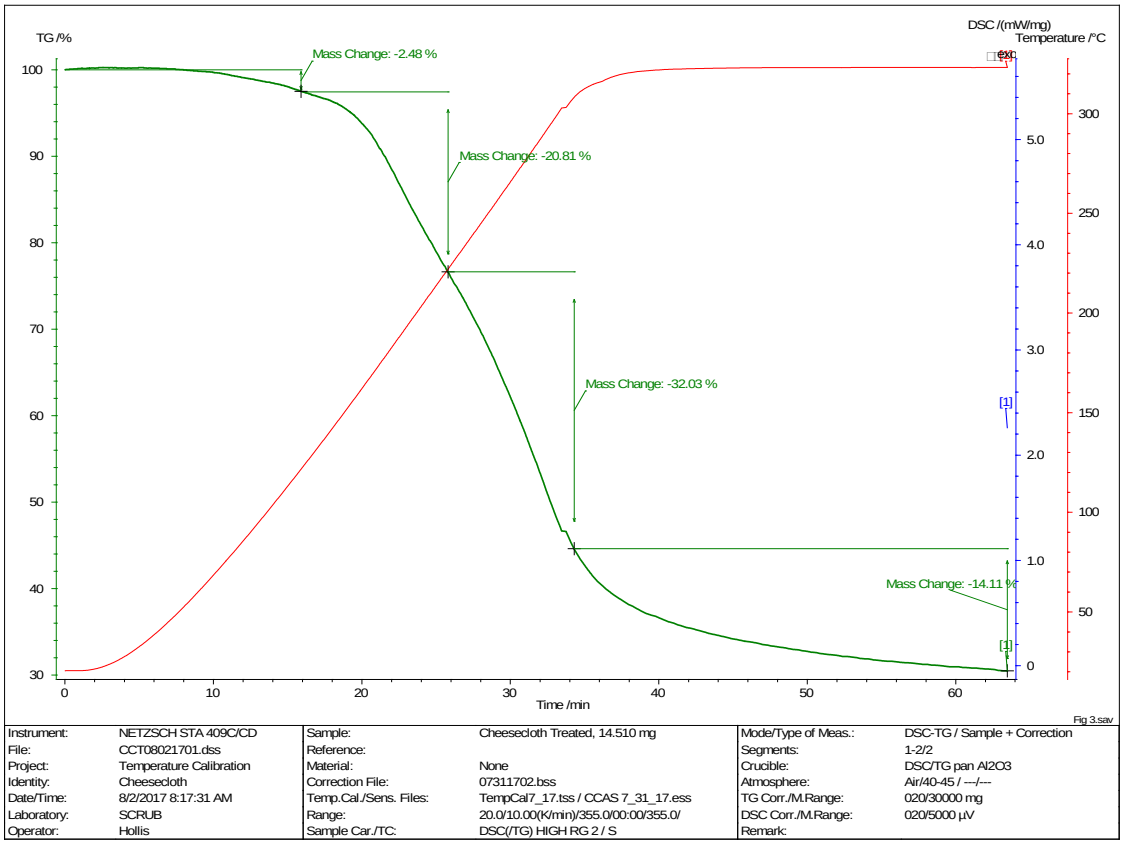
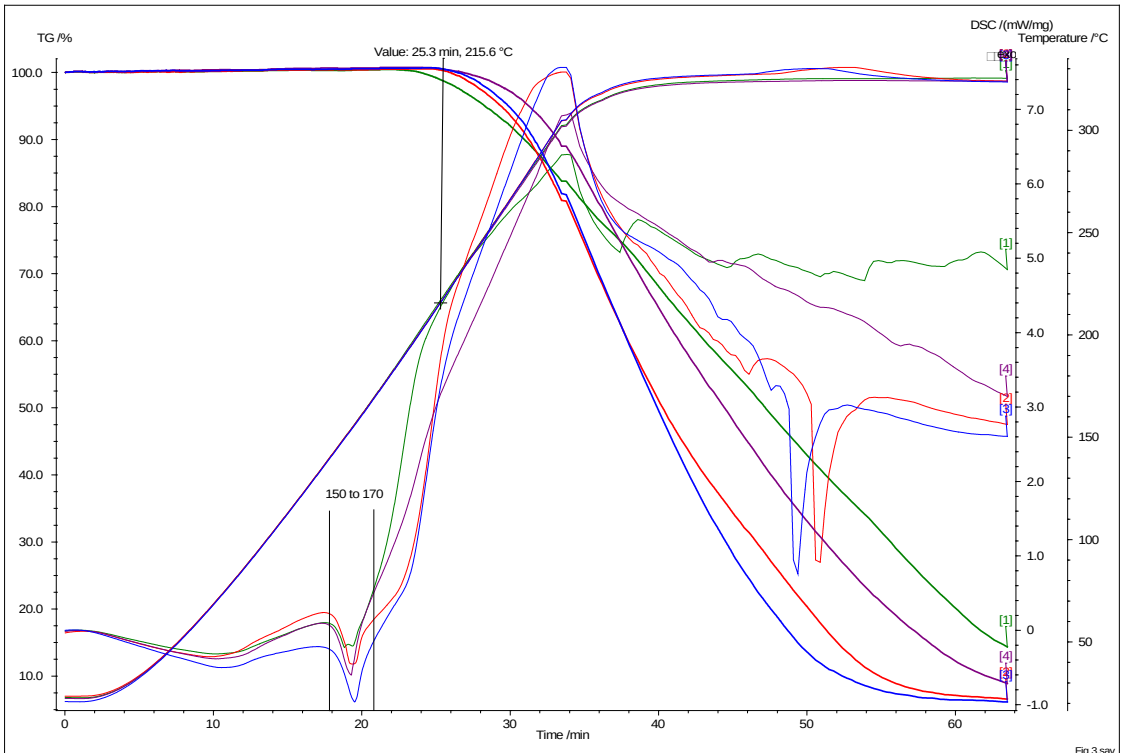


Figure 12. TGA of Treated Cheesecloth (profile 1)

In comparison, the nitric acid treated CC, Figure 12, showed a mass loss at 100°C of 2.45% that is identified as water loss based on CALC: C-CDE-17-001.[23] Thermal oxidation began at 122.5°C and had three distinctive mass loss ranges; 122.5 to 222°C (20.81% mass loss), 222°C to 300°C (32.03% mass loss) and 300°C to 308°C over the 30 min hold (14.11% mass loss). This decrease in the on-set of thermal decomposition from treated and untreated CC was also in previous studies. [23]

The polypropylene samples, Hazmat and Kimtech, had similar TGA/DSC curves for this initial temperature profile, Figure 13. Results for the treated and untreated Hazmat wipes are shown in green and red respectively. Results for the treated and untreated Kimtech wipes are shown in purple and blue respectively.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

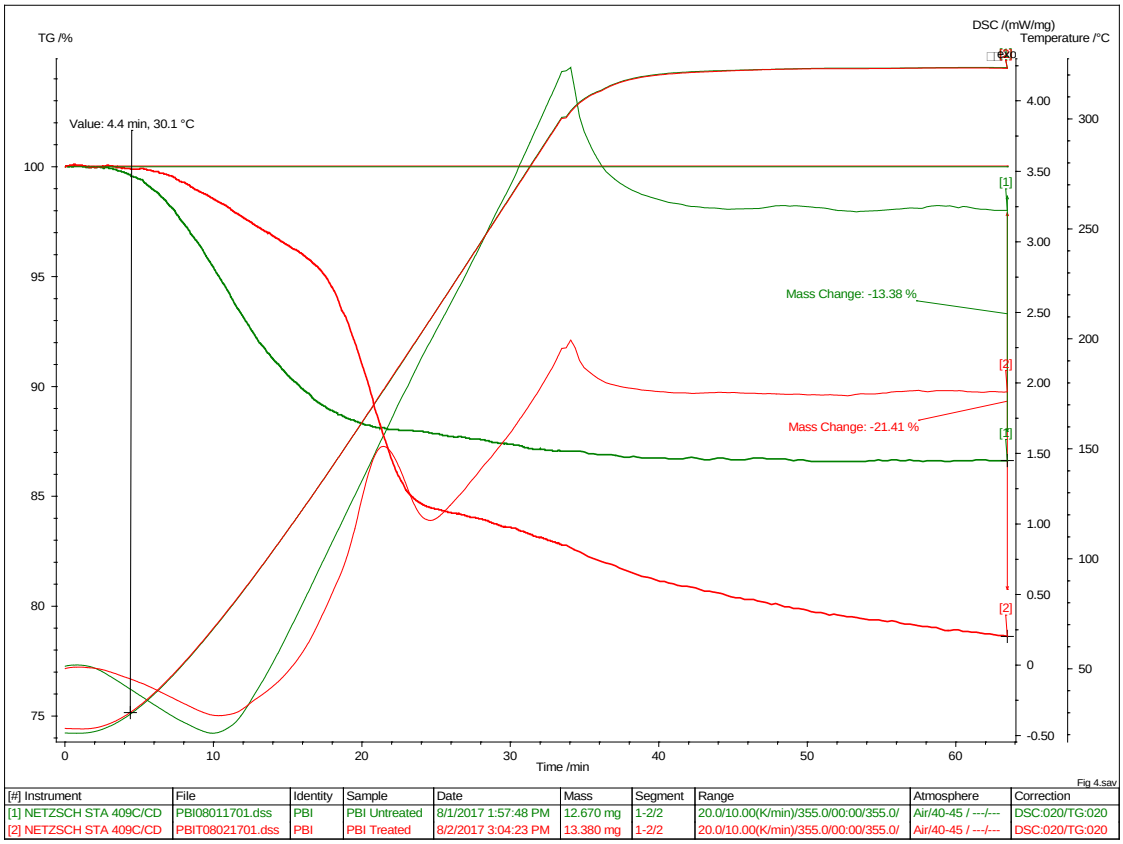


[#] Instrument	File	Identity	Sample	Date	Mass	Segment	Range	Atmosphere	Correction
[1] NETZSCH STA 409C/CD	HZT08021701.dss	Hazmat	Hazmat Treated	8/2/2017 12:50:50 PM	12.690 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020
[2] NETZSCH STA 409C/CD	HZ08011701.dss	Hazmat	Hazmat Untreated	8/1/2017 10:58:17 AM	11.350 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020
[3] NETZSCH STA 409C/CD	KMTC08011701.dss	Kimtech	Kimtech Untreated	8/1/2017 8:48:43 AM	11.760 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020
[4] NETZSCH STA 409C/CD	KMTCT08021701.dss	Kimtech	Kimtech Treated	8/2/2017 10:35:50 AM	10.560 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020

Figure 13. DSC/TGA of Kimtech and Hazmat Wipes (profile 1)

An endotherm was noted, for all treated and untreated polypropylene samples, between 150-170°C, which is associated with the melting point of PP, referenced to fall around 160°C depending on crystallinity and crosslinking of the polymer [24]. A slow rate of thermal oxidation for both the treated and untreated samples began at 215°C for both materials. An approximate 90% mass loss, by the end of the profile, occurred for all sample runs with these two material types.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)



[#] Instrument	File	Identity	Sample	Date	Mass	Segment	Range	Atmosphere	Correction
[1] NETZSCH STA 409C/CD	PBI08011701.dss	PBI	PBI Untreated	8/1/2017 1:57:48 PM	12.670 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020
[2] NETZSCH STA 409C/CD	PBIT08021701.dss	PBI	PBI Treated	8/2/2017 3:04:23 PM	13.380 mg	1-2/2	20.0/10.00(K/min)/355.0/00:00/355.0/	Air/40-45 / ---/---	DSC:020/TG:020

Figure 14 DSC/TGA of PBI (profile 1)

PBI showed a much different result in comparison to the other three material types, as seen in Figure 14. The mass loss for the untreated sample, shown in green, resulted in only a 13% total mass loss. The treated sample, shown in red, has a slight increase of 21% total mass loss. The PBI had the lowest initial onset of mass change of any of the materials starting at 30°C for both samples. This result will be discussed in more detail with the second temperature profile where mass spectral data can be incorporated. The mass loss of the treated samples did show a distinctive 'two staged' distribution (175°C and 300°C), whereas the untreated only began to show mass loss similar to the second exotherm around the 300°C hold temperature.

The second temperature profile, 30-600°C, with no hold, was performed to analyze the results using a DSC calibration. CC showed similar initiation of the thermal oxidation for treated and untreated samples at 120°C and 300°C respectively. The untreated CC, Figure 15, had 2 distinctive exotherms associated with this decomposition. The initial exotherm, starting at 328°C, had an enthalpy of 734.5 J/g. A second exotherm started at 386°C with an enthalpy of 1782 J/g.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

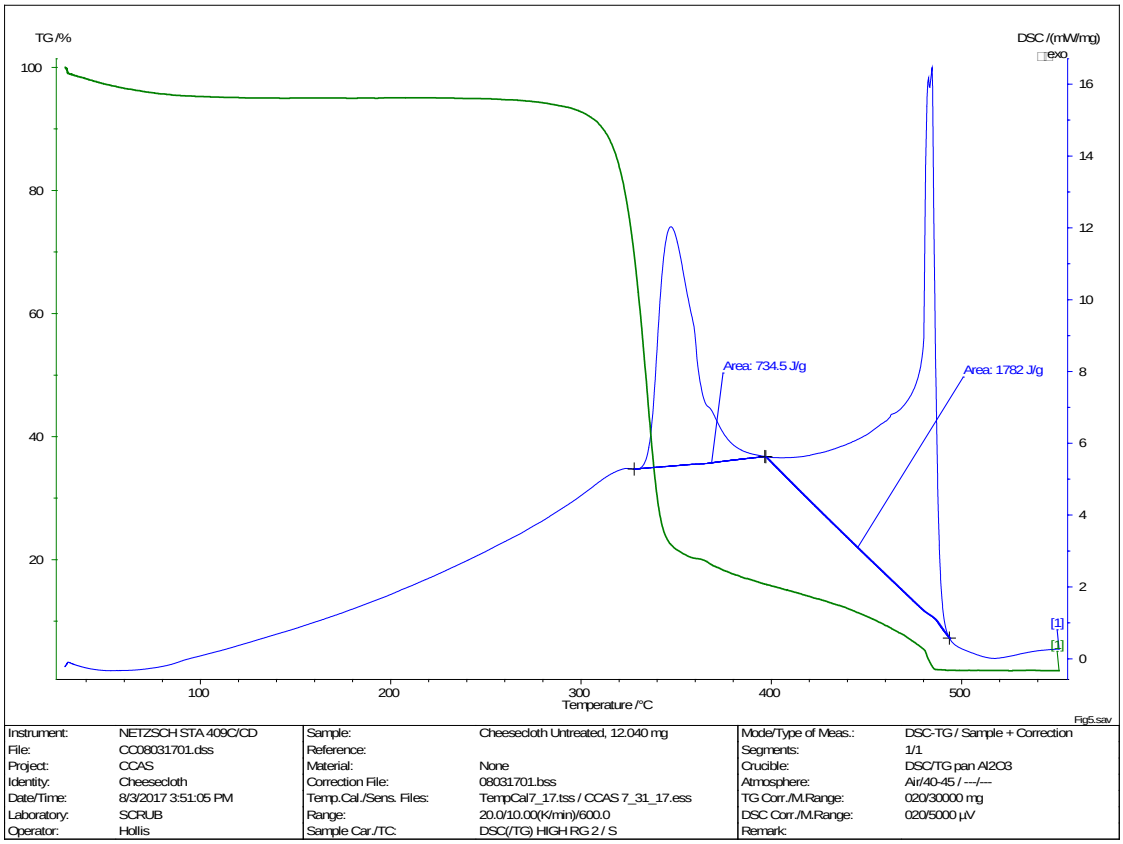


Figure 15. DSC of Untreated Cheesecloth (profile 2)

Unlike the untreated CC shown above, the treated CC did not have distinctive exotherms. However, four exothermic events were identified: 150 to 237°C, 237 to 368°C, 368 to 440°C, and 440 to 600°C, as shown in Figure 16, with a combined enthalpy of 7446 J/g. This enthalpy is approximately three times greater than the enthalpy of the untreated sample.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

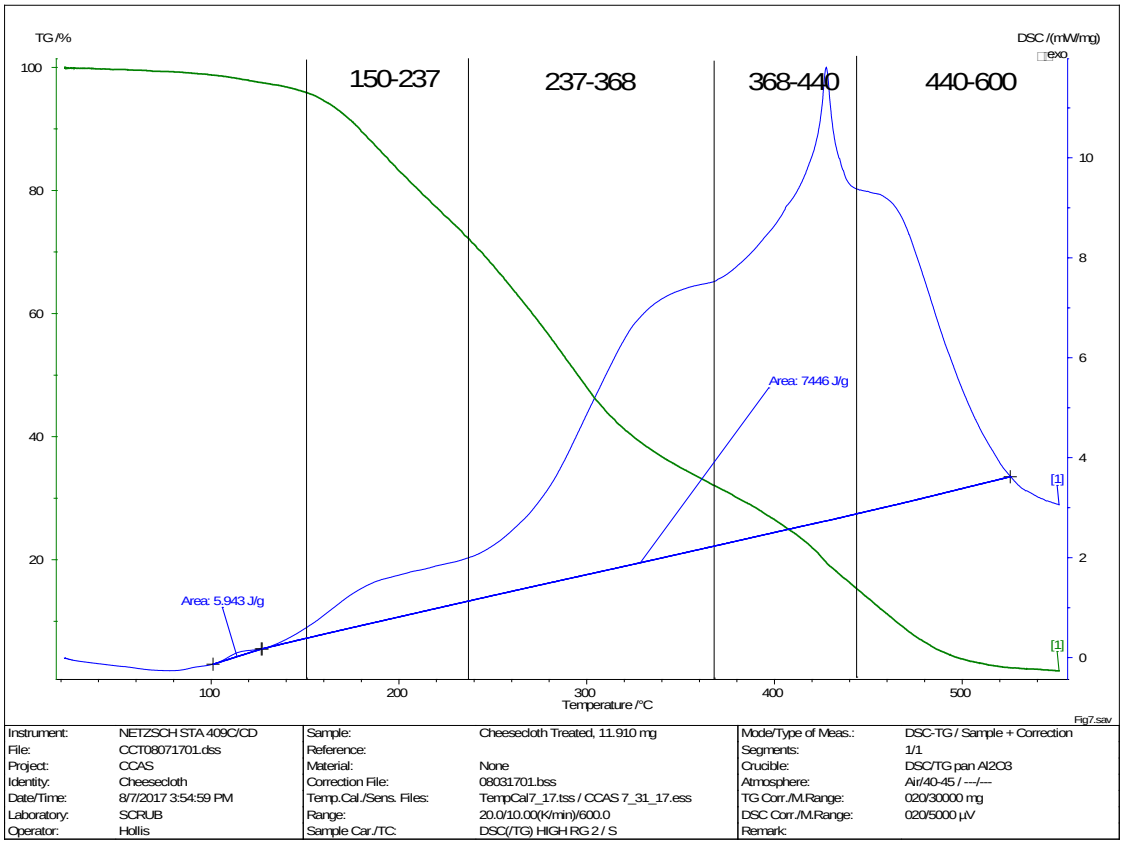
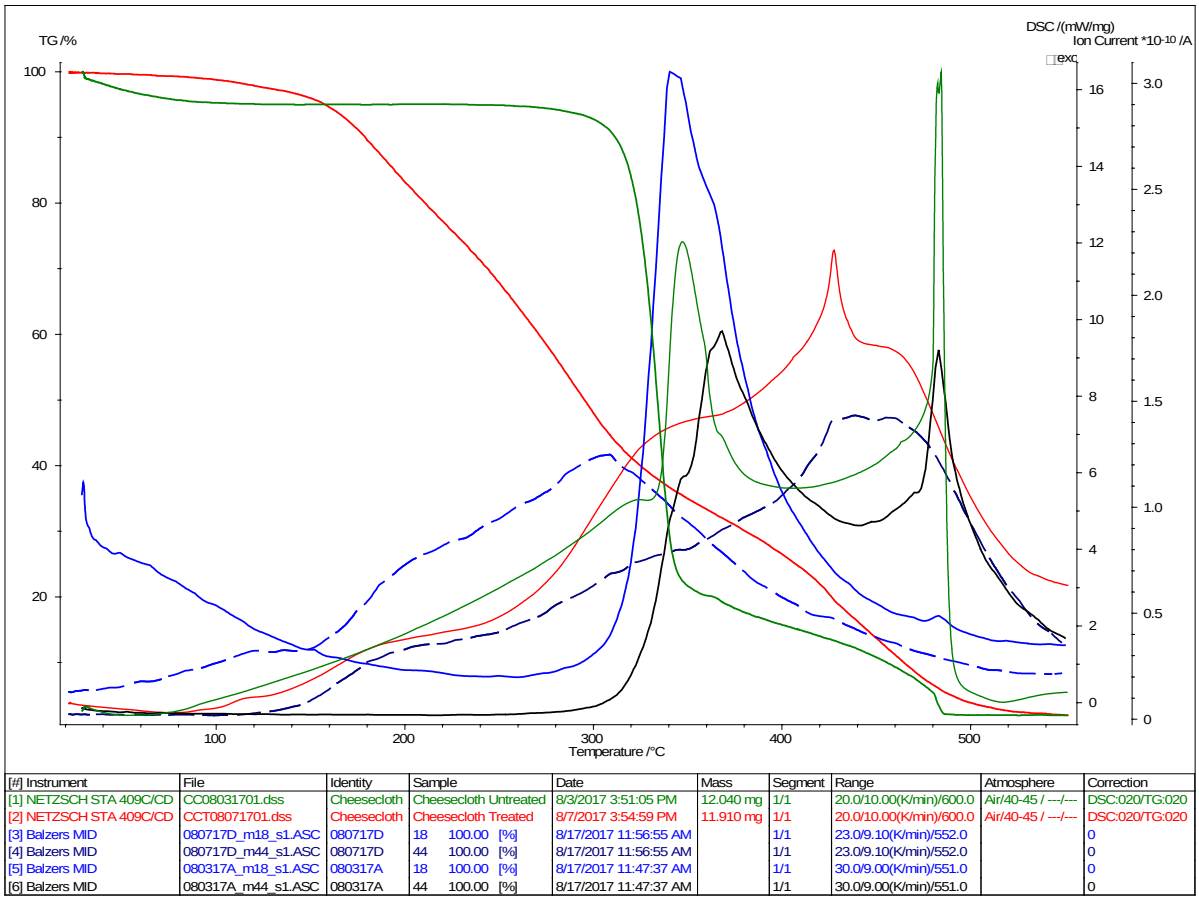


Figure 16. DSC of Treated Cheesecloth (profile 2)

Figure 17 combines the DSC/TGA spectra of both the treated and untreated cheesecloth along with the mass spectral data of both of these events. For the untreated sample, water with a mass-to-charge ratio (m/z) of 18 (blue line) and carbon dioxide with m/z 44 (black line) were detected as expected for a combustion event. Likewise, the primary mass spectral species in the treated CC analysis were water and carbon dioxide (dashed blue and black lines respectively). It is also noted that for the treated sample, at low temperatures the water associated with the combustion is found in high concentrations compared to carbon dioxide but becomes less predominant at high temperatures. This is most likely due to hydrogen consumption at lower temperatures, <400°C, causing the sample to transition to a "charcoal" like material.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)



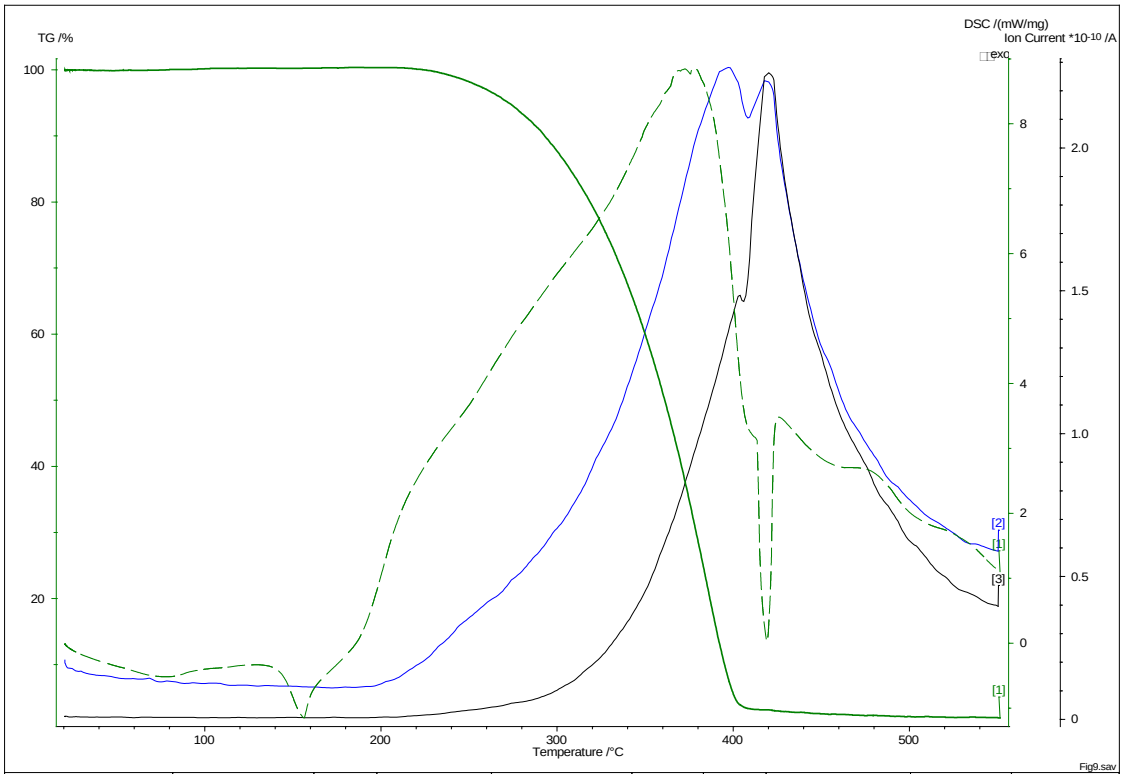
[#] Instrument	File	Identity	Sample	Date	Mass	Segment	Range	Atmosphere	Correction
[1] NETZSCH STA 409C/CD	CC08031701.dss	Cheesecloth	Cheesecloth Untreated	8/3/2017 3:51:05 PM	12.040 mg	1/1	20.0/10.00(K/min)/600.0	Air/40-45 / ---/---	DSC:020/TG:020
[2] NETZSCH STA 409C/CD	CCT08071701.dss	Cheesecloth	Cheesecloth Treated	8/7/2017 3:54:59 PM	11.910 mg	1/1	20.0/10.00(K/min)/600.0	Air/40-45 / ---/---	DSC:020/TG:020
[3] Balzers MID	080717D_m18_s1.ASC	080717D	18 100.00 [%]	8/17/2017 11:56:55 AM		1/1	23.0/9.10(K/min)/552.0		0
[4] Balzers MID	080717D_m44_s1.ASC	080717D	44 100.00 [%]	8/17/2017 11:56:55 AM		1/1	23.0/9.10(K/min)/552.0		0
[5] Balzers MID	080317A_m18_s1.ASC	080317A	18 100.00 [%]	8/17/2017 11:47:37 AM		1/1	30.0/9.00(K/min)/551.0		0
[6] Balzers MID	080317A_m44_s1.ASC	080317A	44 100.00 [%]	8/17/2017 11:47:37 AM		1/1	30.0/9.00(K/min)/551.0		0

Figure 17. DSC with MS of Untreated and Treated Cheesecloth (profile 2)

As seen in the ramp and hold profile in Figure 13, the Hazmat and Kimtech samples had similar results for the second temperature profile. These results are provided in Appendix 3. Initial on-set of thermal oxidation begins at 210°C and complete combustion of the material is seen by 400°C. The endotherm at approximately 155°C is seen again in all samples and as before is referenced to the melting point of PP, giving an enthalpy of 56-61 J/g for Kimtech and 46 J/g for Hazmat. A second endotherm between 400-420°C with an enthalpy ranging from 2409-3724 J/g was seen in both Kimtech samples and the untreated Hazmat. There was no significant mass-loss seen during this event. A reason for this endotherm has not been identified. A small endotherm was also seen in the treated Hazmat but was not as predominant due to a large exotherm at approximately the same temperature and may have been masked. The DSC integrated area for both the Kimtech samples and the untreated Hazmat ranged from 210 to 410°C, resulted in over 90% of the total mass loss, and had an enthalpy of 2860-3384 J/g, which is less than half the value for treated cheesecloth.

The mass loss of both materials began at 192°C. The treated Hazmat showed a slower rate of thermal oxidation at temperatures between 275-300°C. At 275°C the untreated sample mass loss increased rapidly as compared to the treated. The treated sample increased by 20-40°C.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

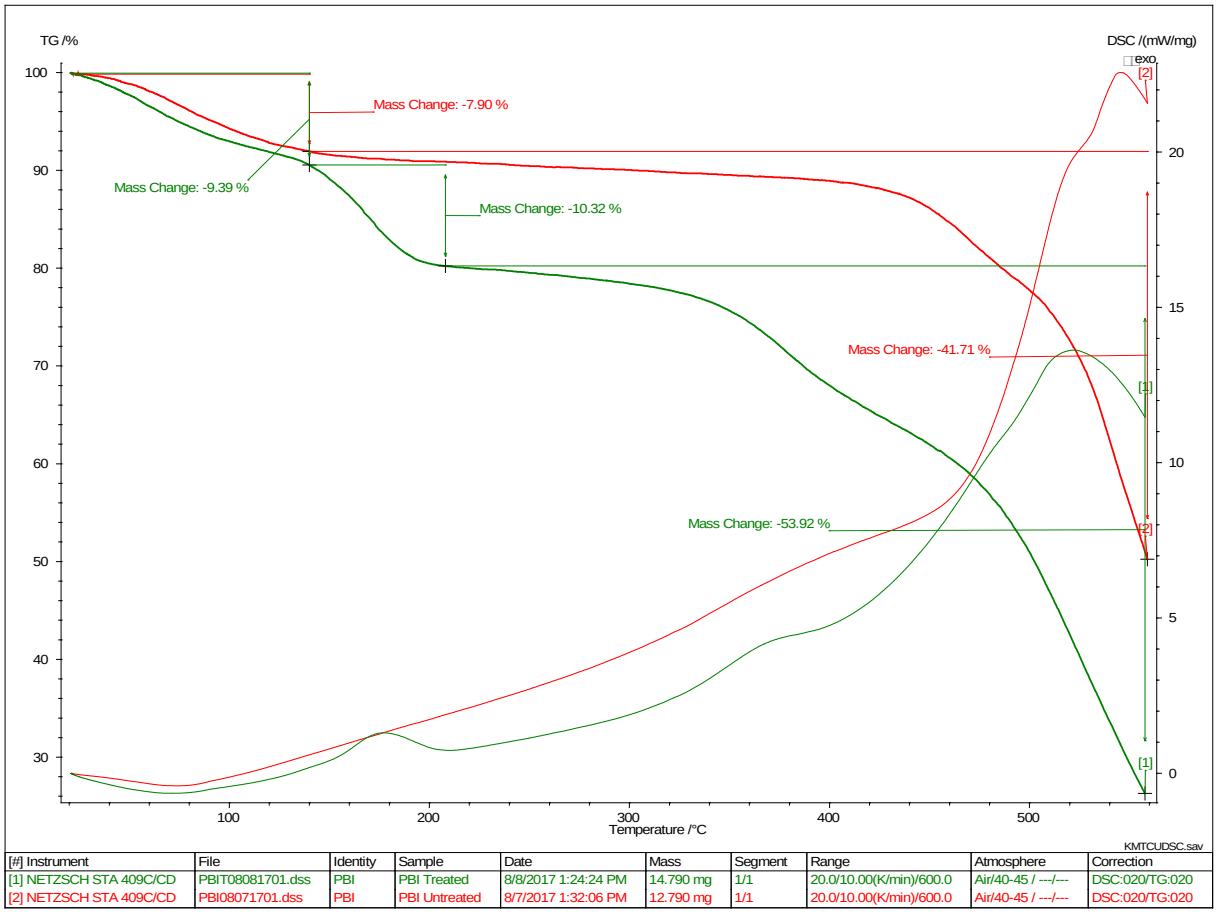


[#] Instrument	File	Identity	Sample	Date	Mass	Segment	Range	Atmosphere	Correction
[1] NETZSCH STA 409C/CD	KMTC08071701.dss	Kimtech	Kimtech Untreated	8/7/2017 8:24:52 AM	12.350 mg	1/1	20.0/10.00(K/min)/600.0	Air/40-45 / ---/---	DSC:020/TG:020
[2] Balzers MID	080717A_m18_s1.ASC	080717A	18 100.00 [%]	8/17/2017 12:03:43 PM		1/1	22.0/9.20(K/min)/552.0		0
[3] Balzers MID	080717A_m44_s1.ASC	080717A	44 100.00 [%]	8/17/2017 12:03:43 PM		1/1	22.0/9.20(K/min)/552.0		0

Figure 18. DSC with MS of Untreated Kimtech (profile 2)

Mass spectral data for the Kimtech and Hazmat are similar to the results seen in the initial thermal oxidation of CC, with water and carbon dioxide being the predominant species. However, the water to carbon dioxide ratio remains consistent throughout the thermal decomposition of the material suggesting that the charcoal material did not form with these samples. Figure 18 represents the untreated Kimtech as an example of the mass spectra collected (blue and black lines are water and carbon dioxide respectively).

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)



[#] Instrument	File	Identity	Sample	Date	Mass	Segment	Range	Atmosphere	Correction
[1] NETZSCH STA 409C/CD	PBIT08081701.dss	PBI	PBI Treated	8/8/2017 1:24:24 PM	14.790 mg	1/1	20.0/10.00(K/min)/600.0	Air/40-45 / ---/---	DSC:020/TG:020
[2] NETZSCH STA 409C/CD	PBI08071701.dss	PBI	PBI Untreated	8/7/2017 1:32:06 PM	12.790 mg	1/1	20.0/10.00(K/min)/600.0	Air/40-45 / ---/---	DSC:020/TG:020

Figure 19. DSC/TGA of Untreated and Treated PBI (profile 2)

The second temperature profile for the PBI again did not completely combust the samples up to the 600°C final temperature, as seen in Figure 19. The untreated material lost roughly 50% during the profile while the treated lost 75%. As seen in the previous profile, similar mass losses of 7.8% to 9.4% were reproduced up to 130°C. The untreated PBI did not show any significant mass loss from 130-400°C. The additional 43% mass loss is seen between 400-600°C, where combustion gases were detected. The treated material did show an exotherm between 130-190°C, with 10% mass loss and an enthalpy of 156.6 J/g. The next mass loss began at 360°C, 40°C less than the untreated, and continued at a similar rate as the untreated for the remaining 57% total mass loss.

Based on mass spectral data shown in Figure 20, the mass loss up to 130°C is associated with interstitial water (dotted blue – H_2O treated, solid blue – H_2O untreated) on the PBI and not thermal oxidation as no carbon dioxide (dotted black – CO_2 treated, solid black – CO_2 untreated) was seen in this region. It was not possible to confirm the presence of residual nitric acid, as proposed in the IR study, because of the difficulty of detecting the highly reactive nitric acid fragments formed by the mass spectrometer. Water and carbon dioxide are seen at all temperatures above 130°C indicating a thermal oxidation process.

6.0 THERMAL GRAVIMETRIC ANALYSIS (TGA) /DIFFERENTIAL SCANNING CALORIMETRY (DSC) (continued)

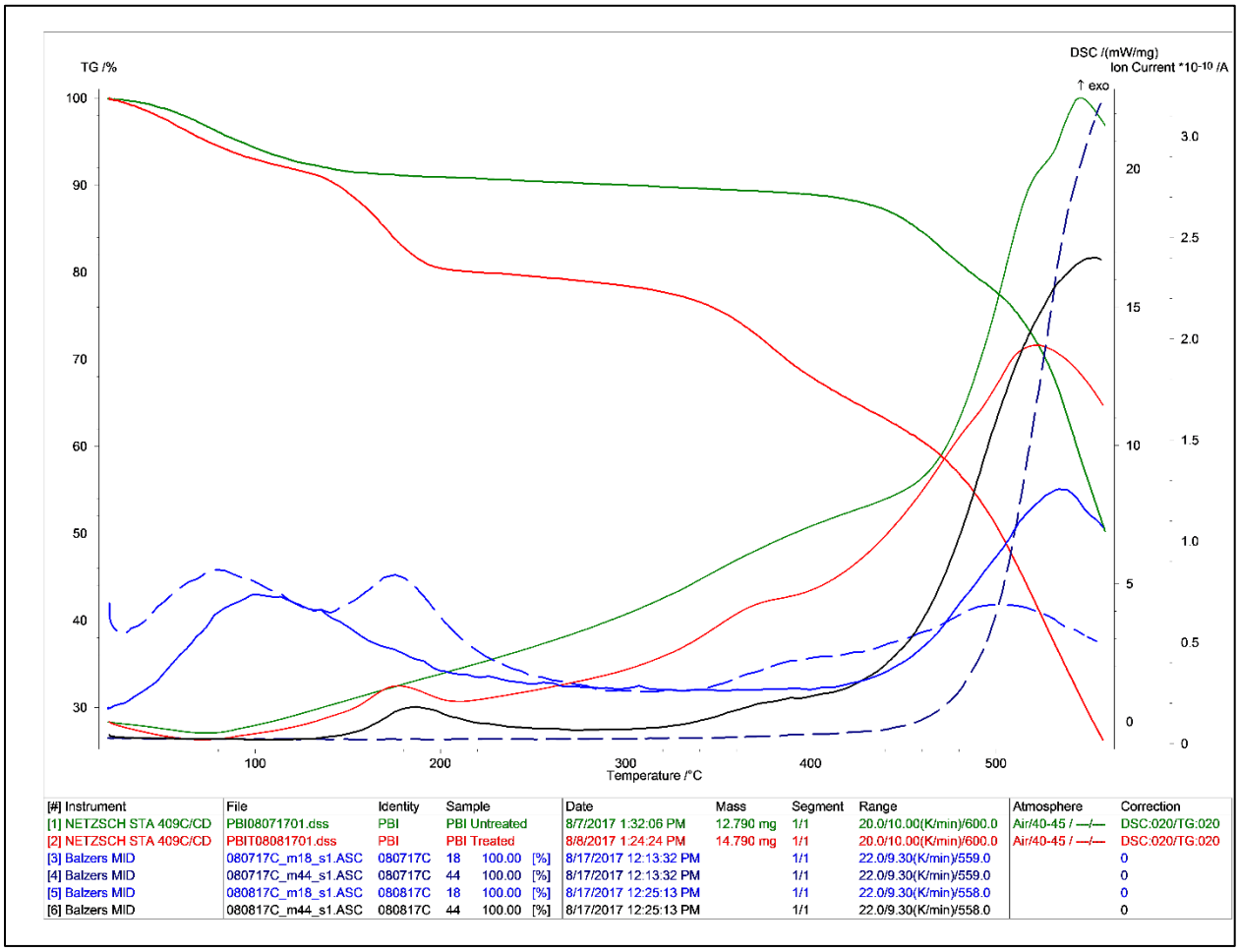


Figure 20. DSC with MS of Untreated and Treated PBI (profile 2)

Table 24. DSC/TGA Scoring

Alternative	Cheese Cloth	Kimtech	PBI	Hazmat
Score	1	3	2	3

Table 25. Scale for Quantitative Scoring

Score	1	2	3
Criteria	Reduction of decomposition temperature by 50 °C or more	Evidence of desorption of HNO_3 and/or H_2O	Same as untreated sample

7.0 CONCLUSION

Based on the results from the initial screening which are scored in Appendix 2 as well as the scoring of the results for the chemical analyses shown in Appendix 4, ranking of the alternatives have been determined. Table 5. Based on this scoring, Kimtech was found to be the best performing alternative, for general housekeeping operations. The other polypropylene wipe tested, Hazmat wipes, did not perform as well because of tearing during use and because a low level contaminant of unknown origin was identified in the samples.

For decontamination of fuel clads, clad surface temperatures can reach several hundred degrees Celsius. Currently, clads are soaked in a decontamination solution containing a diluted 1:2 mixture of nitric acid and HF. Once removed from the decontamination solution, clads are rapidly wiped with water soaked cheesecloth to remove surface contamination. Neither Kimtech nor Hazmat are viable since both melt at approximately 155°C. PBI is the proposed alternative at high temperatures although it performs poorly for absorption. Absorption is less of an issue in this process than for housekeeping. Further studies of PBI are required prior to implementation to ensure that the material meets the requirements for minimal contamination required for contact with clads and to ensure that clads can be handled safely with this material.

Table 26. Ranking of Cheesecloth Alternatives

Alternative	Usability Score	Chemical Analysis Score	Total Score	Ranking
Cheese Cloth	3.5	1.6	5.1	4
Kimtech	4.75	2.8	7.55	1
PBI	4	2.0	6	3
Hazmat	4.75	2.6	7.35	2
Chamois	2.75	NA	NA	NA

8.0 APPENDICES AND ATTACHMENTS

Appendix	Title
1	*Screening Details and Results*
2	*Qualitative Scoring (Including Screening Evaluation)*
3	*Chemical Analysis Details and Results*

Attachment	Title
A	*None*

Appendix 1, Screening Details and Results

^{238}Pu Radiation Tolerance

For ^{238}Pu radiation tolerance screening, samples of each of the alternatives and cheesecloth were cut into approximately 3 inch squares. These samples were placed in zip lock bags and introduced into the dropbox. The bag were unsealed and taped to the wall of the drop box adjacent to the aqueous processing glovebox at a height of approximately 5.5 ft. so they were not easily contaminated or disturbed when items were moved through the dropbox. An electronic personal dosimeter was taped on the dropbox wall along with the sample to measure the cumulative dose the samples received. After 42 days the batteries on the EPD died. The day before, the EPD read approximately 9000 mRem. Shortly after battery failure the room was evacuated for several days so immediate battery replacement was not possible. Photos of the samples were made on day 5 and 29 but are not shown as no changes were observed.

Durability Screening

Prior to the sample testing, 5 batches of sodium chloride (NaCl or salt), the solid "spill" surrogate were poured on the floor of an unused glovebox. The salt batches were slightly wetted with water to make a paste and were allowed to dry for 1 week to form a hard crust on the stainless steel surface. Cheesecloth, Hazmat pads, and PBI were cut in approximately 12 inch squares to match the approximate size of the Kimtech and chamois wipes.

Each of the samples was tested on a separate salt spill wetted with water and then used to wipe up approximately one third of the "spill". Then a second operator rinsed and wrung out the sample and used it to clean up another third of the salt spill. Observations were made independently by each operator about ease of cleaned up, whether the material remained intact, whether the material could be wrung out and re-wetted, etc.

Absorbency Screening

To determine absorbency, samples of each of the alternatives and cheesecloth were cut into approximately 4 in^2 squares. A petri dish was weighed (m_1) and then a sample of each alternative was added to the container and weighed (m_2). The sample was removed from the petri dish and placed into another container where concentrated nitric acid (15.8M) was pipetted onto the sample until it was saturated as evidenced by standing acid in the container. Then the sample was moved back to the original petri dish and the weight (m_3) was measured again. The mass of the liquid absorbed by the sample (m_A) was calculated by:

$$m_A = m_3 - m_2 - m_1$$

The absorbency factor (A_f) (wet to dry ratio) was calculated by dividing the mass of the absorbed liquid (m_A) by the mass of the sample ($m_2 - m_1$)

$$A_f = m_A/(m_2 - m_1)$$

The results from the absorbency screening are shown in Table 6 and the raw data are provided in Table 7.

Table 27. Absorbency Factors for Different Acid Molarities

Molarity	Cheesecloth	Chamois	Hazmat	Kimtech	PBI
15.8	4.39	0.87	11.85	6.32	1.60
10	6.58	NA	10.76	5.87	1.32
5	5.03	1.42	9.80	5.23	1.12
1	6.06	3.01	8.79	4.45	1.07

Table 28. Absorption Testing Results

Cheesecloth Alternatives - Absorbtion Study Data (6/19/2017)

Cheesecloth

M	PD (g)	DRY (g)	SAMPLE (g)	WET (g)	WET-DRY (g)	Liquid (g)	%	Wet/ Dry Ratio
15.8	54.54	59.52	4.98	86.35	26.83	21.85	438.69	4.39
10	50.82	54.2	3.38	79.82	25.62	22.24	657.87	6.58
5	57	59.52	2.52	74.71	15.19	12.67	502.86	5.03
1	50.92	53.56	2.64	72.20	18.64	16.00	606.17	6.06

Chamois

M	PD (g)	DRY (g)	SAMPLE (g)	WET (g)	WET-DRY (g)	Liquid (g)	%	Wet/Dry Ratio
15.8	54.39	56.44	2.05	60.27	3.83	1.78	86.88	0.87
10	55.71	55.75	0.04	61.80	6.05	6.01	15012.50	150.13
5	58.56	60.51	1.95	65.24	4.73	2.78	142.46	1.42
1	55.25	57.15	1.9	64.78	7.63	5.73	301.47	3.01

Hazmat

M	PD (g)	DRY (g)	SAMPLE (g)	WET (g)	WET-DRY (g)	Liquid (g)	%	Wet/Dry Ratio
15.8	45.18	47.48	2.3	77.02	29.54	27.24	1184.52	11.85
10	57.05	59.34	2.29	86.26	26.92	24.63	1075.55	10.76
5	54.16	56.14	1.98	77.53	21.39	19.41	980.10	9.80
1	56.64	58.67	2.03	78.54	19.87	17.84	878.72	8.79

Kimtech

M	PD (g)	DRY (g)	SAMPLE (g)	WET (g)	WET-DRY (g)	Liquid (g)	%	Wet/Dry Ratio
15.8	56.35	57.17	0.82	63.17	6.00	5.18	631.83	6.32
10	51.99	52.78	0.79	58.20	5.42	4.63	586.58	5.87
5	57.73	58.51	0.78	63.37	4.86	4.08	522.82	5.23
1	56.14	56.87	0.73	60.85	3.98	3.25	444.66	4.45

PBI

M	PD (g)	DRY (g)	SAMPLE (g)	WET (g)	WET-DRY (g)	Liquid (g)	%	Wet/Dry Ratio
15.8	54.51	56.15	1.64	60.42	4.27	2.63	160.24	1.60
10	48.27	50	1.73	54.02	4.02	2.29	132.31	1.32
5	49.66	51.4	1.74	55.09	3.69	1.95	112.01	1.12
1	50.63	52.32	1.69	55.81	3.49	1.80	106.57	1.07

M	Molarity	DRY	Weight of Petri Dish and 1 folded sample
PD	Petri Dish	WET	Weight of Petri Dish and 1 saturated sample
		SAMPLE	Weight of dry sample

Integrity in Nitric Acid Screening

Samples from the absorbency screening were then placed in a well ventilated fume hood and were observed and photographed after 2 days, 3 days, and 25 days. Photos from day 2 and day 25 are shown in Figures 21-60 to show the changes that occurred over time.

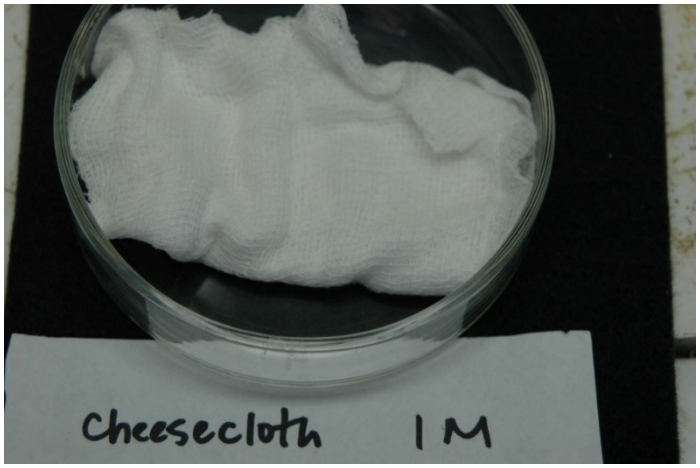


Figure 21. Cheesecloth exposed to 1M HNO_3 for 2 days

Figure 23. Cheesecloth exposed to 5M HNO_3 for 2 days

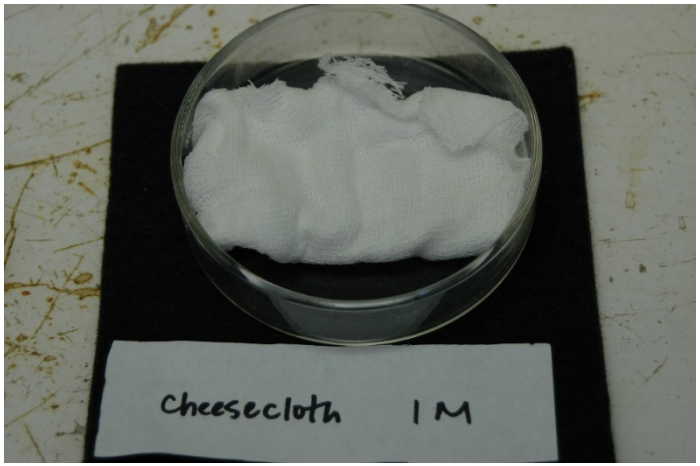


Figure 22. Cheesecloth exposed to 1M HNO_3 for 25 days

Figure 24. Cheesecloth exposed to 5M HNO_3 for 25 days

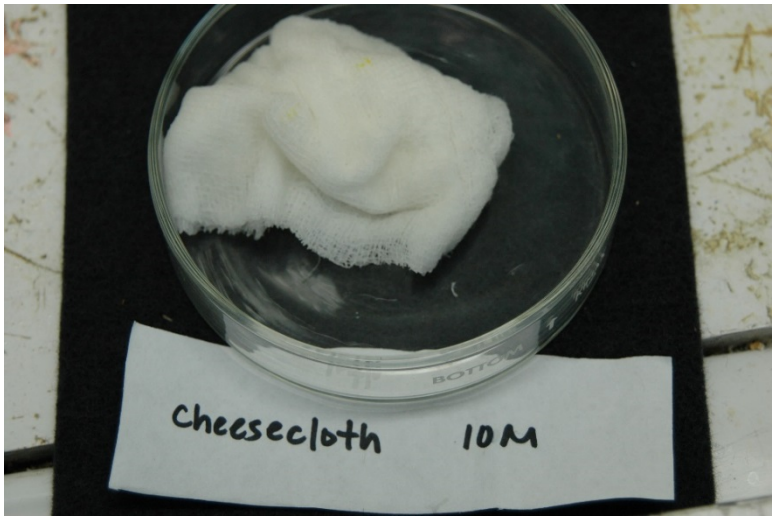


Figure 25. Cheesecloth exposed to 10M HNO_3 for 2 days

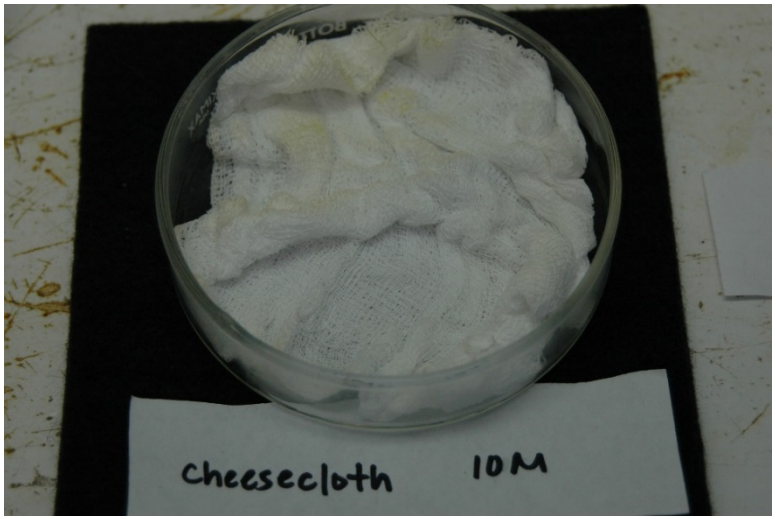


Figure 26. Cheesecloth exposed to 10M HNO_3 for 25 days

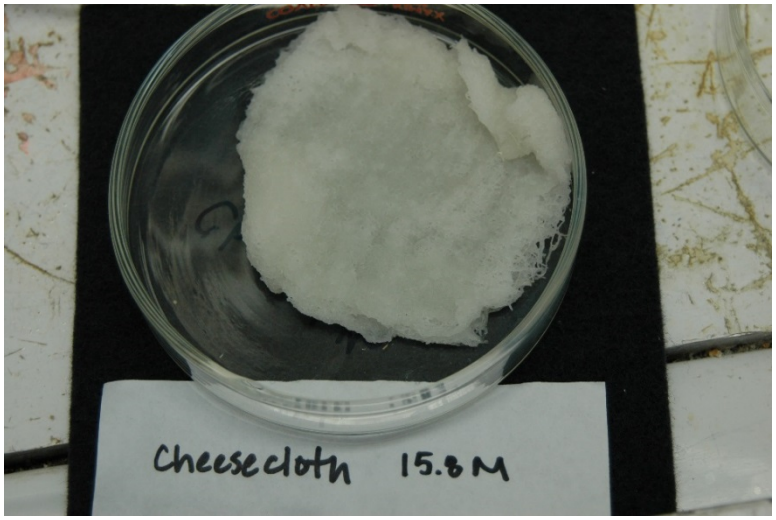


Figure 27. Cheesecloth exposed to 158M HNO_3 for 2 days

Figure 28. Cheesecloth exposed to 15.8M HNO_3 for 25 days

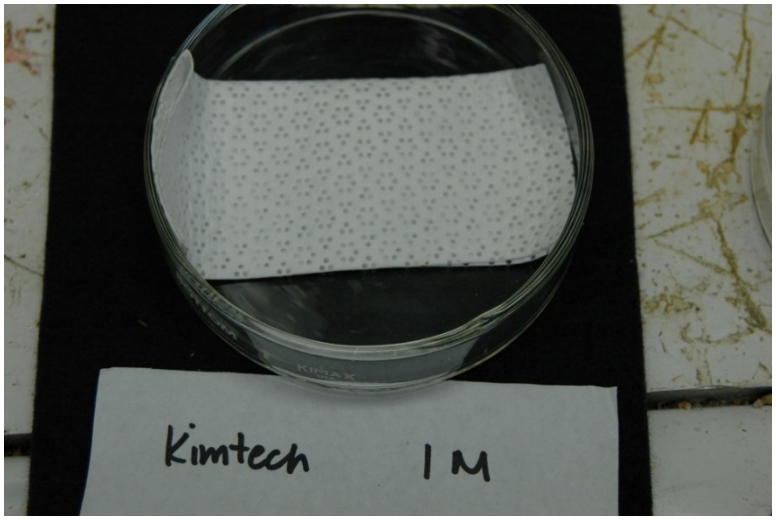


Figure 29. Kimtech exposed to 1M HNO_3 for 2 days

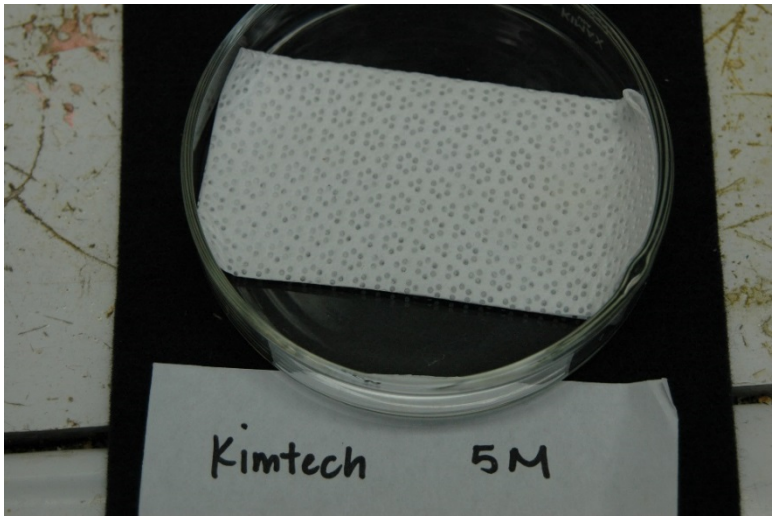


Figure 31. Kimtech exposed to 5M HNO_3 for 2 days

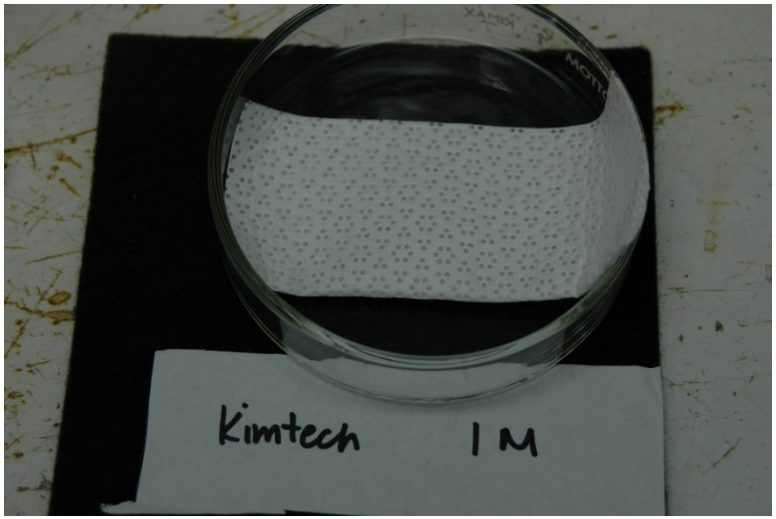


Figure 30. Kimtech exposed to 1M HNO_3 for 25 days

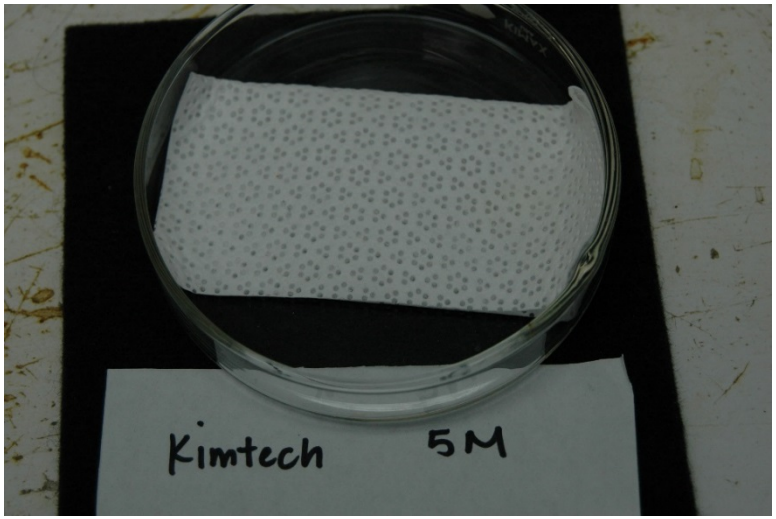


Figure 32. Kimtech exposed to 5M HNO_3 for 25 days

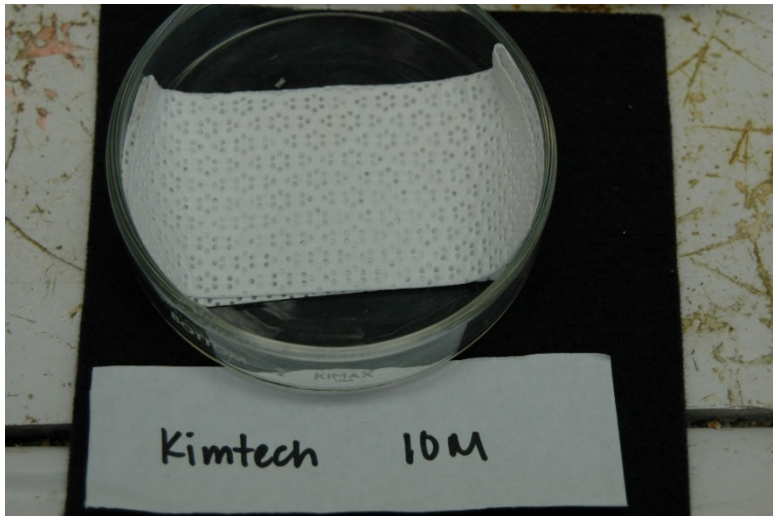


Figure 33. Kimtech exposed to 10M HNO_3 for 2 days

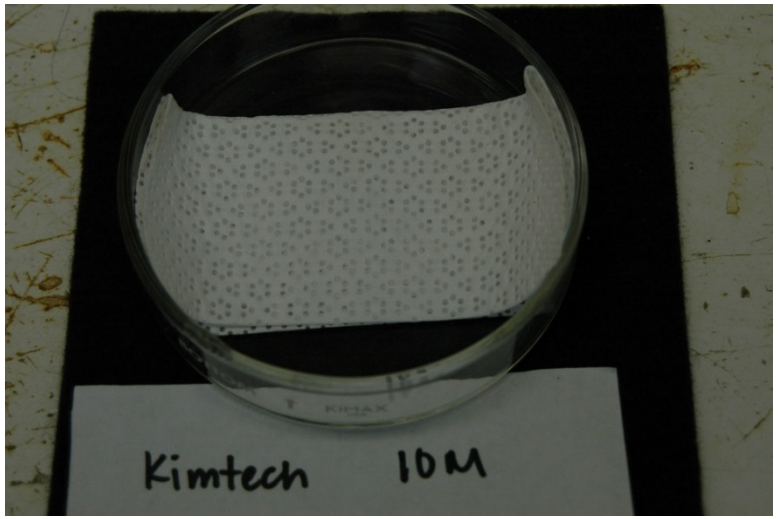


Figure 34. Kimtech exposed to 10M HNO_3 for 25 days

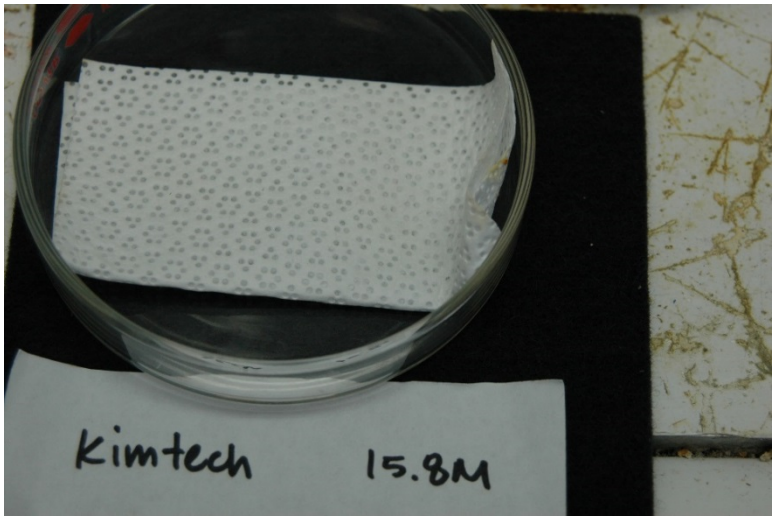


Figure 35. Kimtech exposed to 15.8M HNO_3 for 2 days

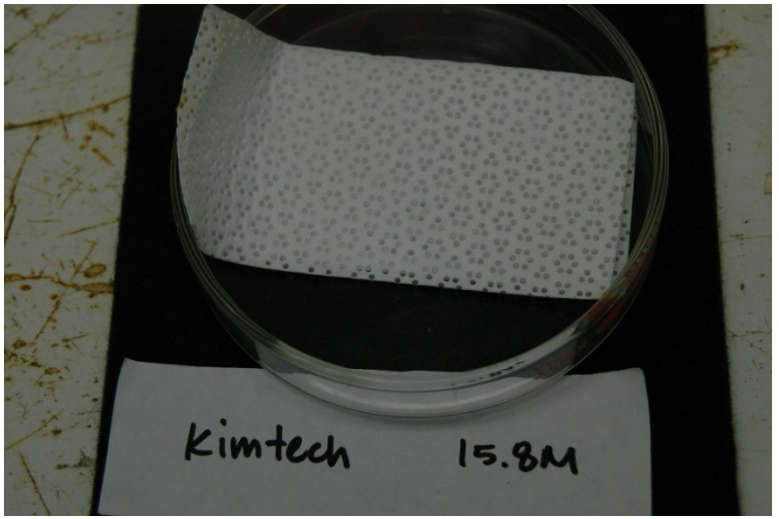


Figure 36. Kimtech exposed to 15.8M HNO_3 for 25 days

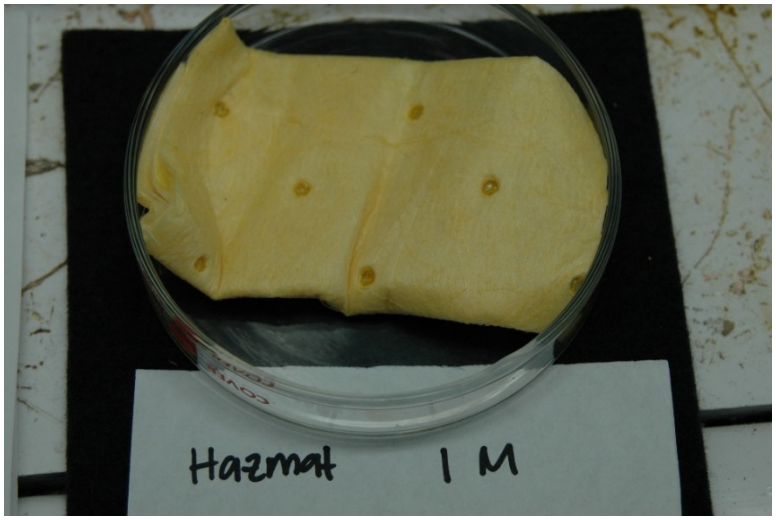


Figure 37. Hazmat exposed to 1M HNO_3 for 2 days

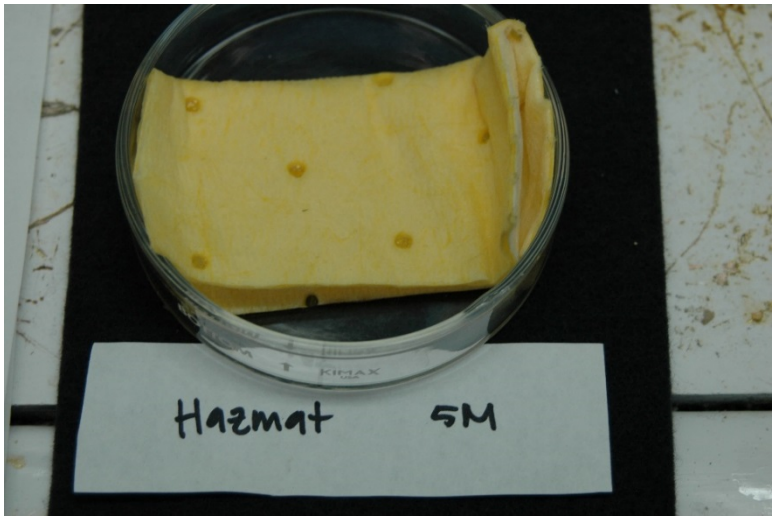


Figure 39. Hazmat exposed to 5M HNO_3 for 2 days

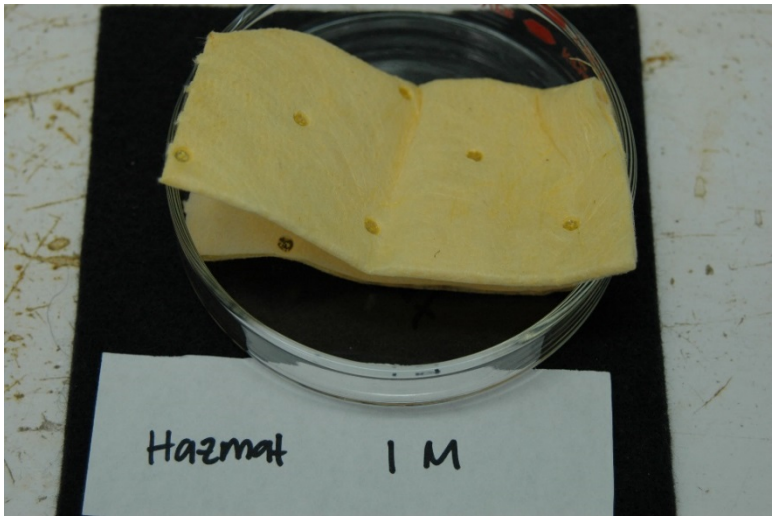


Figure 38. Hazmat exposed to 1M HNO_3 for 25 days

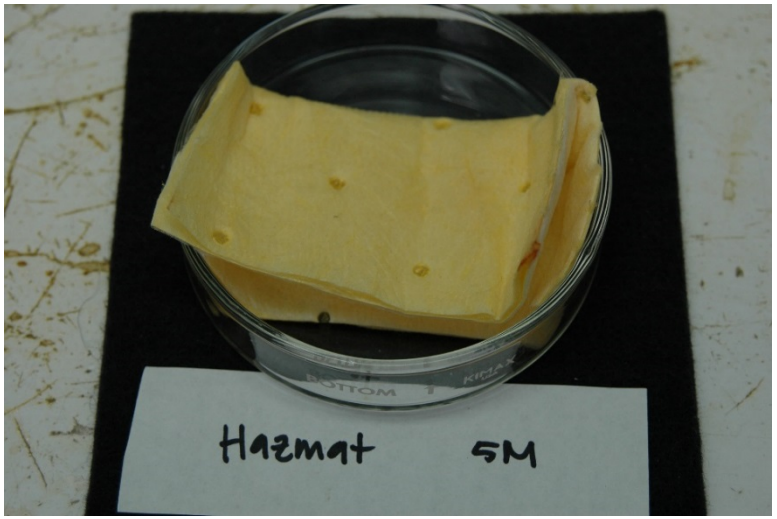


Figure 40. Hazmat exposed to 5M HNO_3 for 25 days

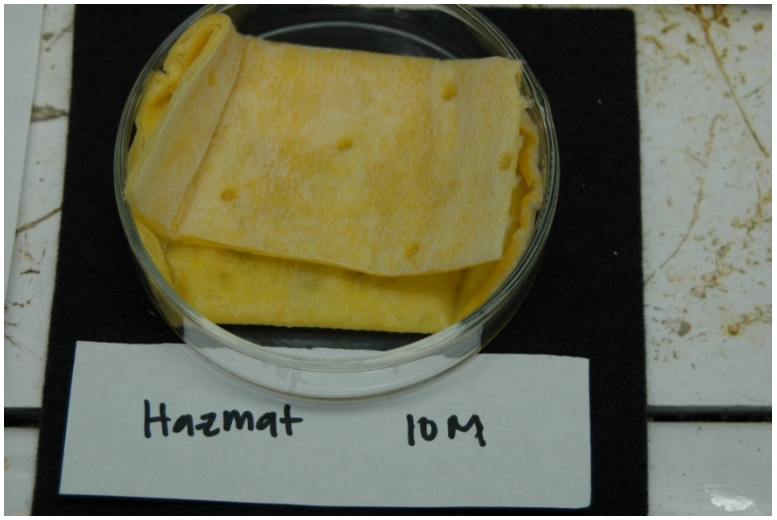


Figure 41. Hazmat exposed to 10M HNO_3 for 2 days

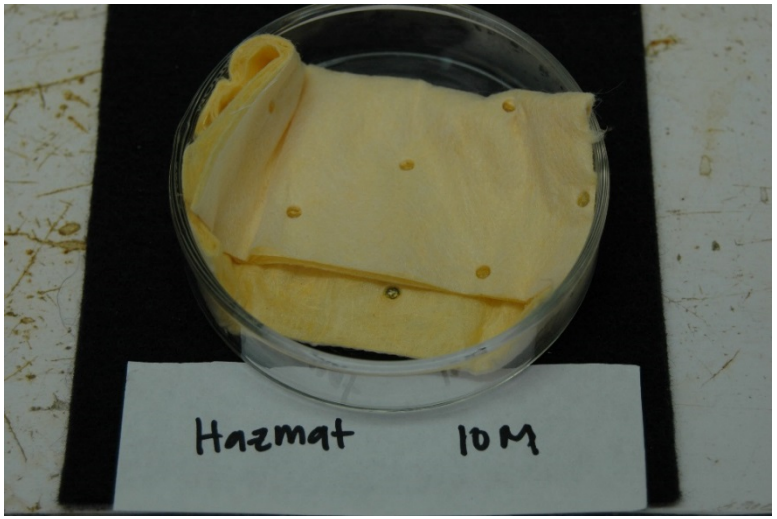


Figure 42. Hazmat exposed to 10M HNO_3 for 25 days

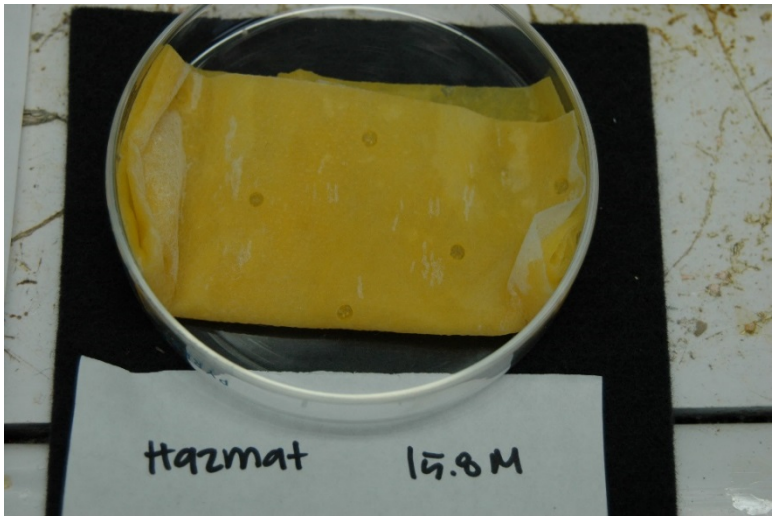


Figure 43. Hazmat exposed to 15.8M HNO_3 for 2 days

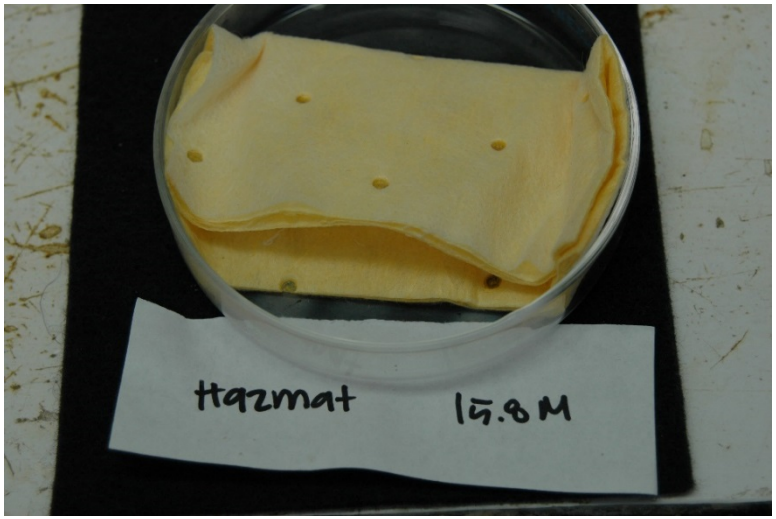


Figure 44. Hazmat exposed to 15.8M HNO_3 for 25 days

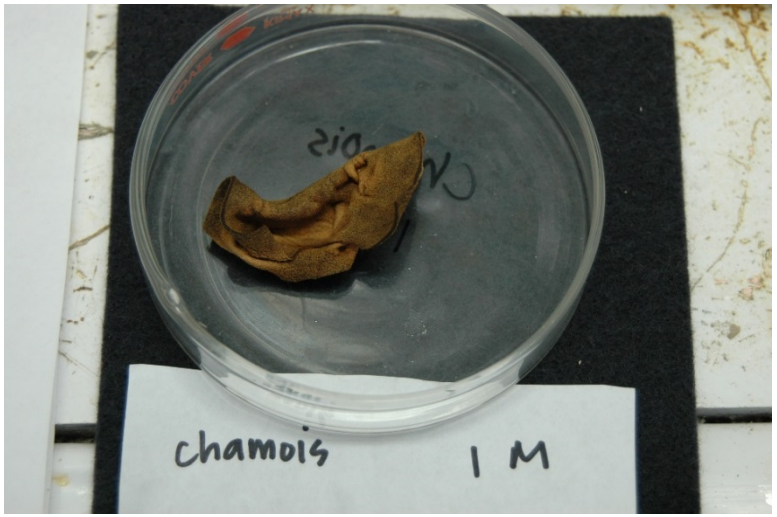


Figure 45. Chamois exposed to 1M HNO_3 for 2 days

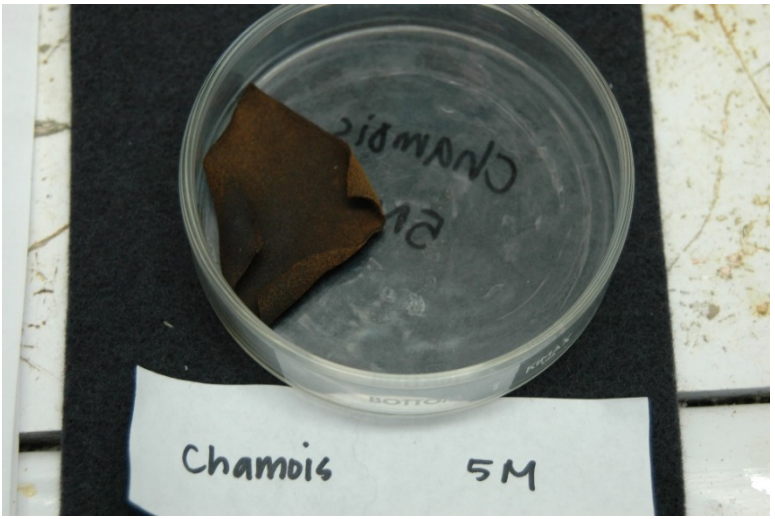


Figure 47. Chamois exposed to 5M HNO_3 for 2 days

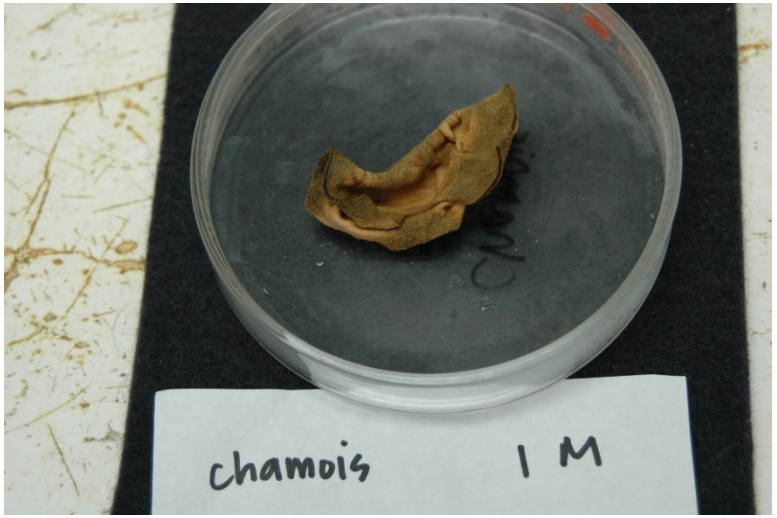


Figure 46. Chamois exposed to 1M HNO_3 for 25 days

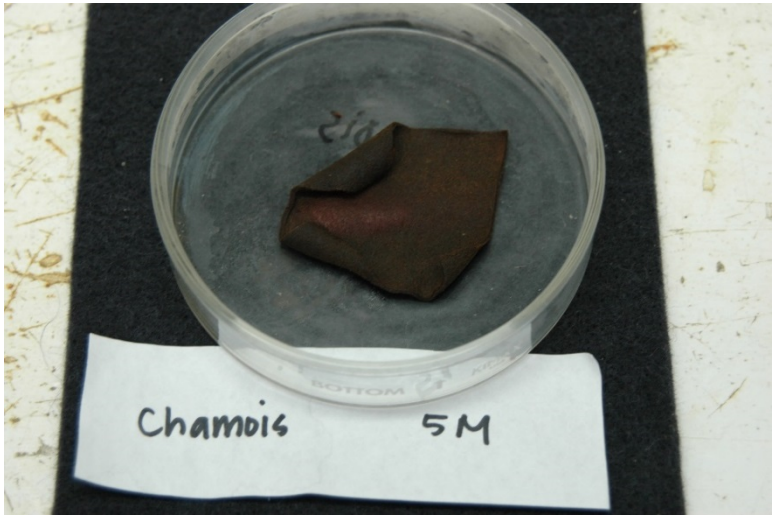


Figure 48. Chamois exposed to 5M HNO_3 for 25 days

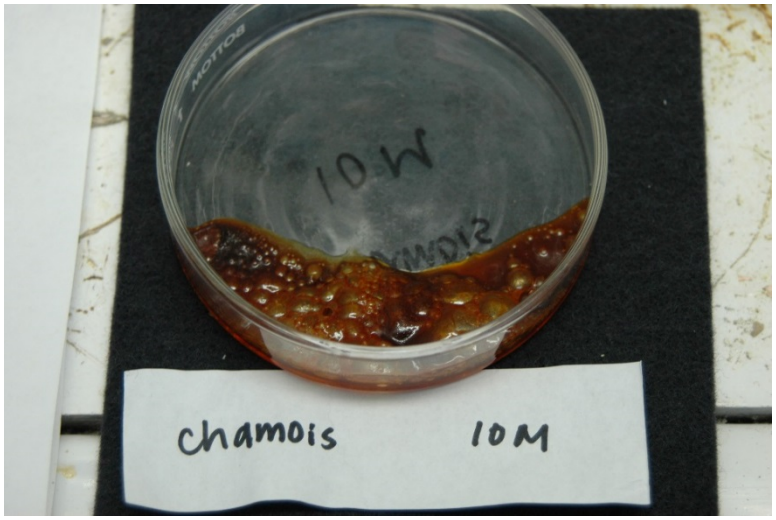


Figure 49. Chamois exposed to 10M HNO_3 for 2 days

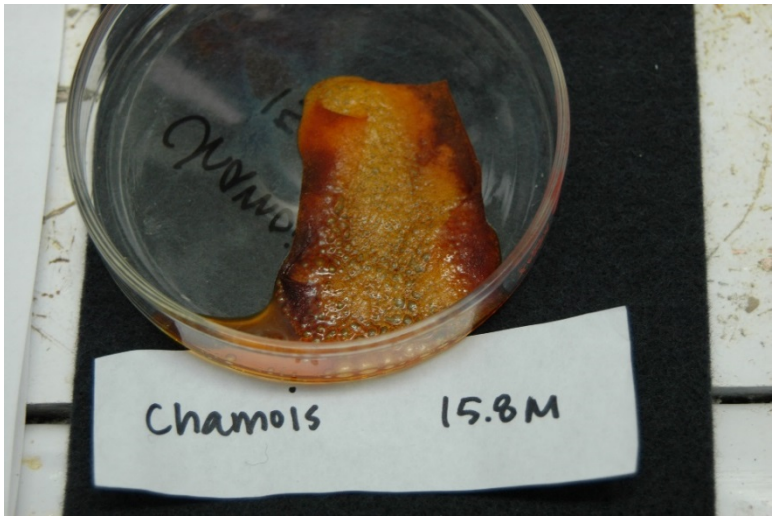


Figure 51. Chamois exposed to 15.8M HNO_3 for 2 days

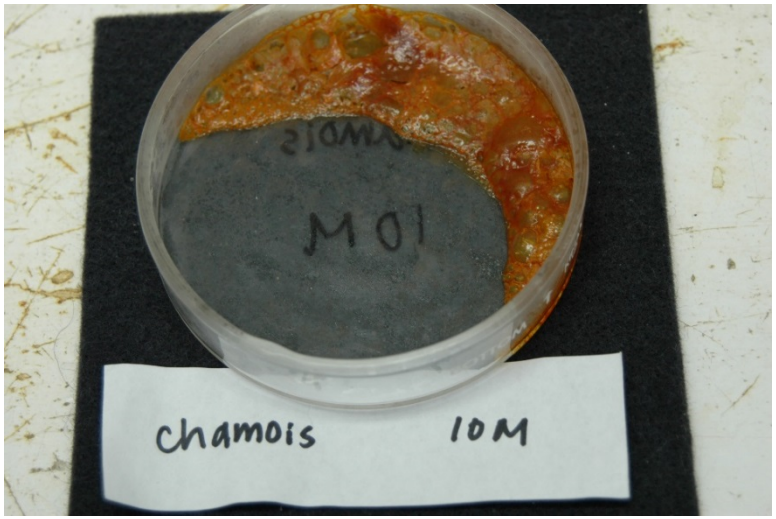


Figure 50. Chamois exposed to 10M HNO_3 for 25 days

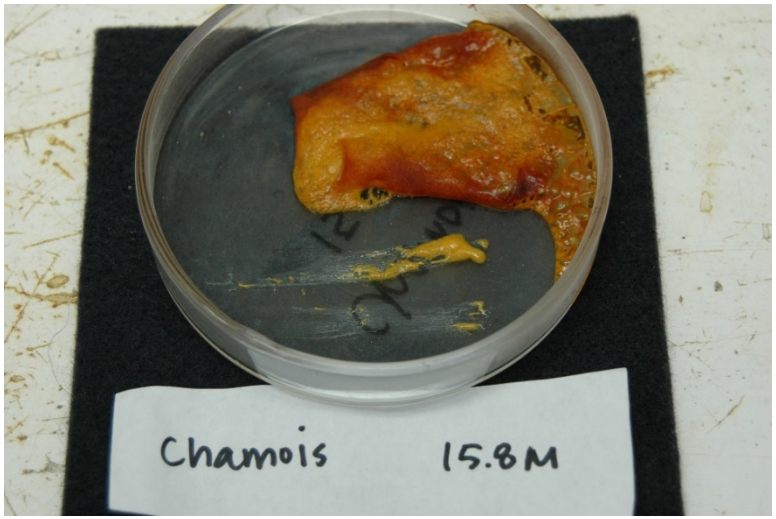


Figure 52. Chamois exposed to 15.8M HNO_3 for 25 days

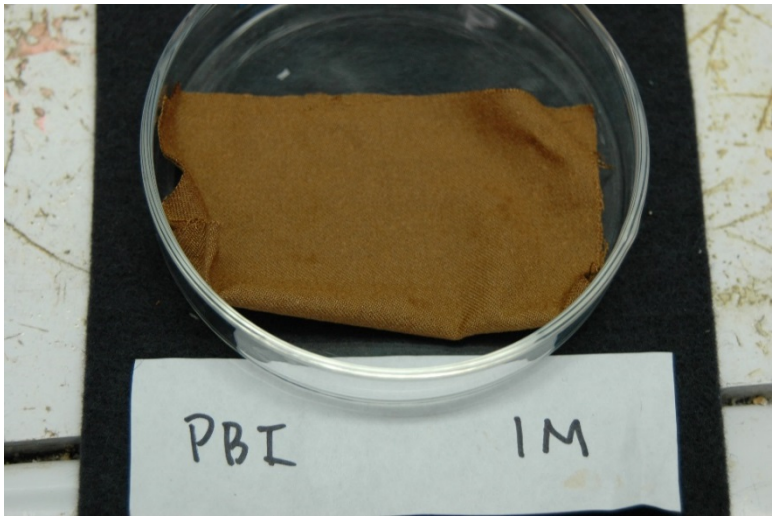


Figure 53. PBI exposed to 1M HNO_3 for 2 days

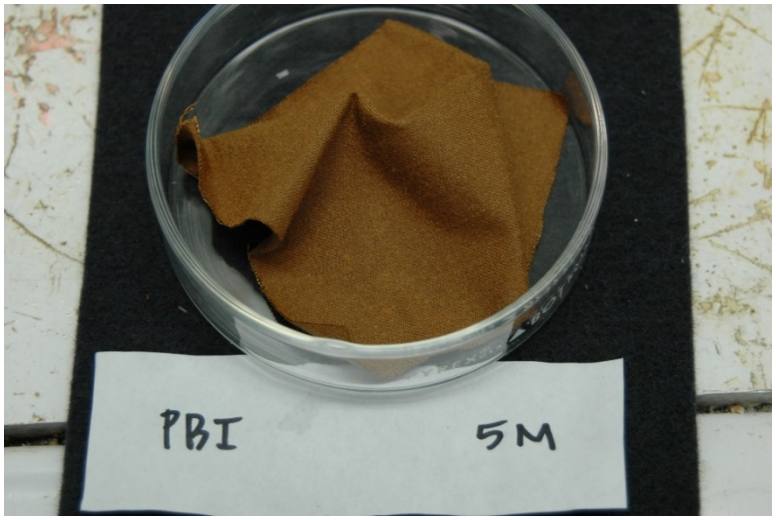


Figure 55. PBI exposed to 5M HNO_3 for 2 days

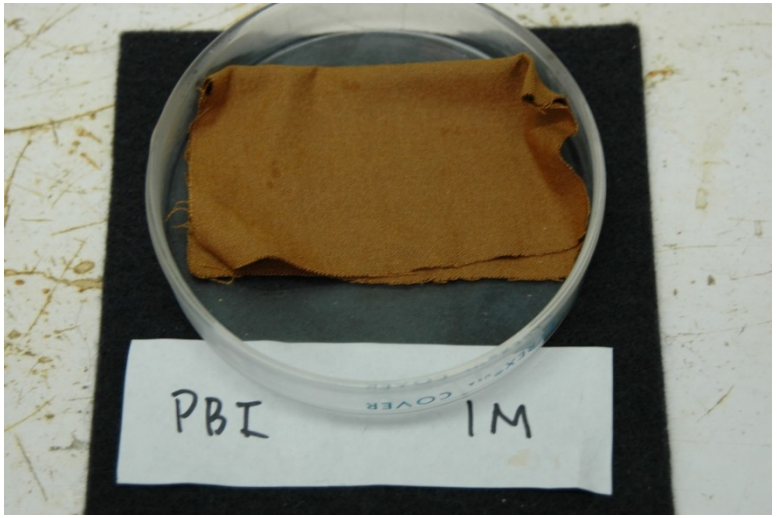


Figure 54. PBI exposed to 1M HNO_3 for 25 days

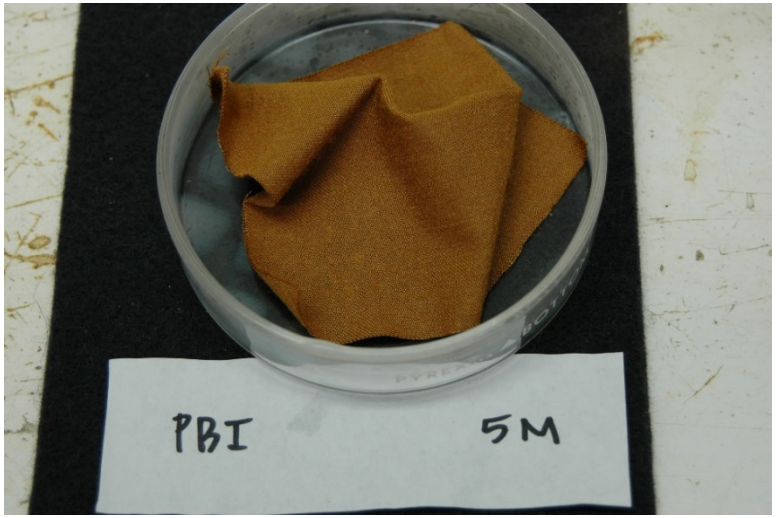


Figure 56. PBI exposed to 5M HNO_3 for 25 days

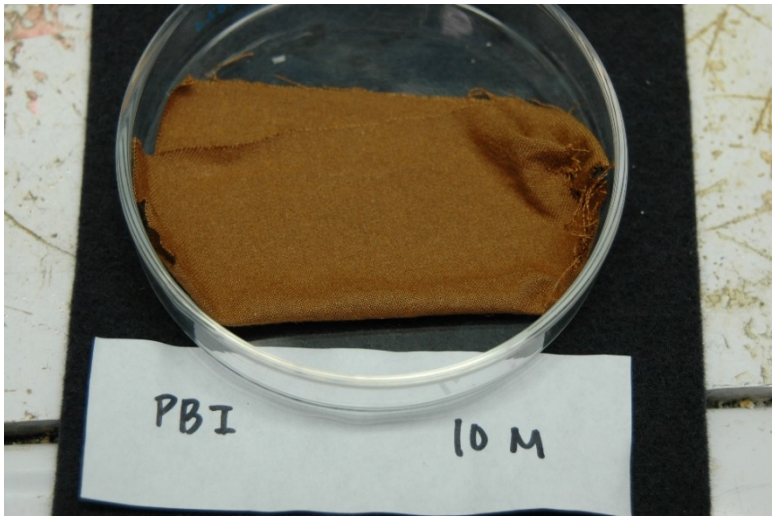


Figure 57. PBI exposed to 10M HNO_3 for 2 days

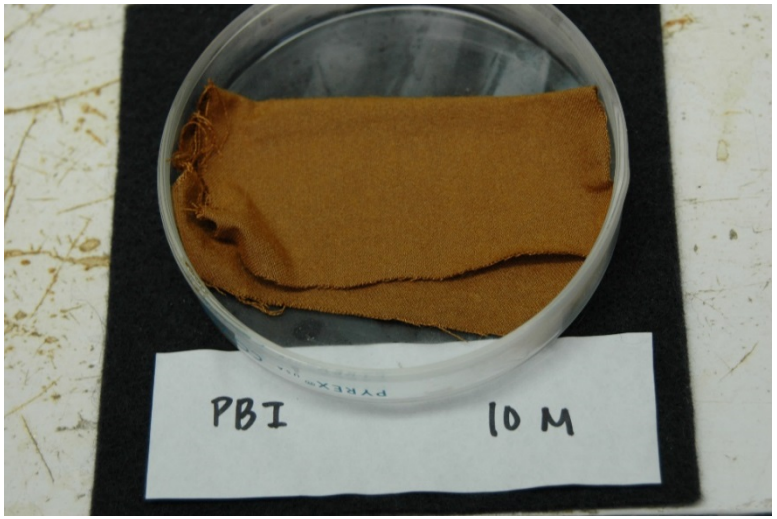


Figure 58. PBI exposed to 10M HNO_3 for 25 days

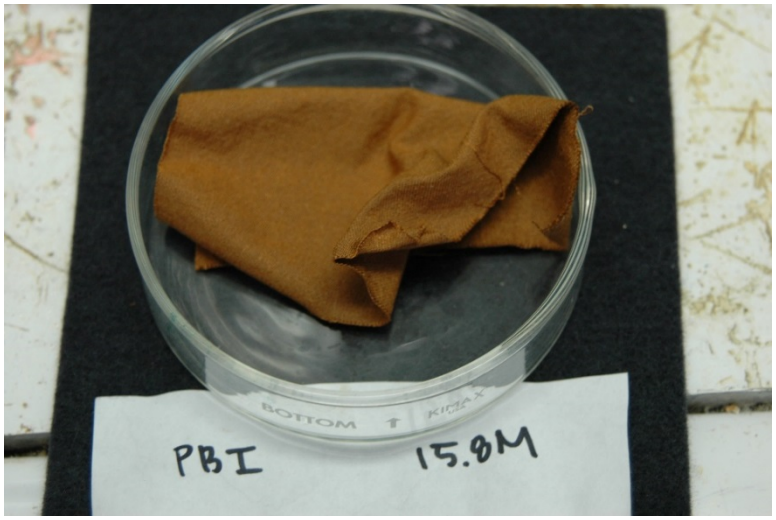


Figure 59. PBI exposed to 15.8M HNO_3 for 2 days

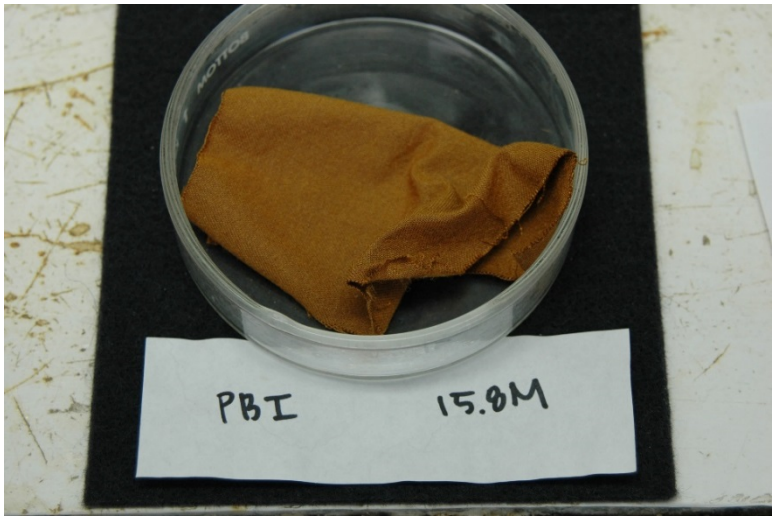


Figure 60. PBI exposed to 15.8M HNO_3 for 25 days

Appendix 2, Qualitative Scoring (Including Screening Evaluation)

Table 29. Scoring for Alternatives Exposed to 1M HNO_3

Alternative	Rad Tolerance	Absorbency	Integrity in HNO_3	Durability	Sub-Total	Qual Score: Sub-Total/3
Cheese Cloth	5	4	4	5	13	4.3
Kimtech Pure W4 Wipers	5	3	5	4	13	4.3
PBI Products	5	1	5	5	11	3.7
Hazmat Sorbent SM Pad, Premium	5	5	5	5	15	5.0
Chamois	5	2	2	4	9	3.0

Table 30. Scoring for Alternatives Exposed to 5M HNO_3

Alternative	Rad Tolerance	Absorbency	Integrity in HNO_3	Durability	Sub-Total	Qual Score: Sub-Total/3
Cheese Cloth	5	3	4	5	12	4.0
Kimtech Pure W4 Wipers	5	3	5	4	13	4.3
PBI Products	5	1	5	5	11	3.7
Hazmat Sorbent SM Pad, Premium	5	5	5	5	15	5.0
Chamois	5	1	2	4	8	2.7

Appendix 2, Qualitative Scoring (Including Screening Evaluation)

Table 31. Scoring for Alternatives Exposed to 10M HNO_3

Alternative	Rad Tolerance	Absorbency	Integrity in HNO_3	Durability	Sub-Total	Qual Score: Sub-Total/3
Cheese Cloth	5	4	4	5	13	4.3
Kimtech Pure W4 Wipers	5	3	5	4	13	4.3
PBI Products	5	1	5	5	11	3.7
Hazmat Sorbent SM Pad, Premium	5	5	5	5	15	5.0
Chamois	5	1*	1	4	7	2.3

Table 32. Scoring for Alternatives Exposed to 15.8M HNO_3

Alternative	Rad Tolerance	Absorbency	Integrity in HNO_3	Durability	Sub-Total	Qual Score: Sub-Total/3
Cheese Cloth	5	3	1	5	9	3.0
Kimtech Pure W4 Wipers	5	4	5	4	14	4.7
PBI Products	5	1	5	5	11	3.7
Hazmat Sorbent SM Pad, Premium	5	5	5	4	13	4.3
Chamois	5	1	1	4	7	2.3

Table 33. Scale for Usability Scoring

Score	1	2	3	4	5
Absorbency (A_f)	<2	≥2	≥4	≥ 6	≥ 8
Integrity	Decomposition	Charring/ Shriveling	Stiffness	Yellowing	No observed changes
Durability	Tears on initial wiping	Cannot wring out material	Tears during use after rinsing/ wringing	Less effective after rinsing/ wringing	No observed change

Appendix 3, Chemical Analysis Techniques Details and Results

Samples for chemical analysis were prepared by cutting small pieces of sample that were small enough to fit in the bottom of a glass sample vial. A sample of each alternative was weighed and placed in a vial. Using the absorbency factor determined during absorbency testing, the amount of 15.8M acid needed to saturate the sample was calculated. This volume of acid was measured out using a micropipette and was added to the sample in the vial. Then the sample was sealed and reweighed. In total, three samples of each alternative were prepared using this technique.

The sample were then placed in an oven at 65 °C and were heated until they all had reached a constant mass over 2 consecutive 4 hour drying intervals. Constant mass was not achieved for all samples on the first day but the masses the following morning were consistent with the masses of the previous afternoon. After that, the samples were place in a desiccator when not being used.

Volatile Organic Carbon (VOC) Off-gas Analysis

Samples of the untreated and HNO_3 treated cloths were analyzed for volatile organic compounds (VOC) off-gas products using static headspace extraction with gas chromatography couple to quadrupole mass spectrometry (HS-GC-qMS). Cloth samples were placed in 20 mL crimp-top headspace vials, saturated with concentrated HNO_3, and sealed with PTFE-backed silicone septa. The headspace extraction unit was operated with temperatures of 60 °C in the vial oven, 150 °C in the sample loop oven, and 200 °C in the transfer line. Helium was used as the pressurization (set to 20 psig) and carrier gas. The sample extraction timing were 20 minutes heated equilibration, 0.20 minutes pressurization, 0.20 minutes sample loop fill, 0.05 minutes sample loop equilibration, and 1.00 minutes sample transfer. The autosampler was set for overlapping operation with a GC cycle time of 37 minutes to extract the subsequent sample while current sample was undergoing analysis. The gas chromatograph was operated using helium carrier gas at 1.4 mL/min with a 20:1 split (turning on the gas saver flow of 15 mL/min at 5.00 minutes). The oven was held at 40 °C for 1 minute, then ramped at 8.5 °C/min to 250 °C, which was held for 1 minute. The zone temperatures were 250 °C at the inlet, 260 °C in the MS transfer line, 230 °C at the ionization source, and 150 °C in the quadrupoles. The mass spectrometer was set to collect *m/z* 34-300, with an ion count threshold of 100 and 2 scans averaged per recorded spectrum.

After completing the sample acquisition, the chromatograms were inspected for chromatographic peaks indicative of VOC off-gas products. The peaks present were integrated using the ChemStation integrator and matched to library spectra using the NIST05 mass spectral database.

Volatile Organic Compounds (VOC) Off-gas Analysis (continued)

No detectable chromatographic peaks were found for the analyses of the untreated cheesecloth or the three alternatives. For all of the samples treated with nitric acid, there were two abundant peaks around 3.6 minutes. These peaks are attributed to nitrogen dioxide (NO_2) which was produced by the thermal decomposition of HNO_3 and from HNO_3 that was transferred to the GC and then converted to NO_2 during the temperature ramp. For the Hazmat pads, there was also a peak at 9.9 minutes for all three Hazmat sample runs. Mass spectrometry software tentatively identified this substance as 3,3-diethyl-pentane by comparison with the National Institute of Standards and Technology (NIST) mass spectral library with a q value of 40. The q value is a probability assignment that the unknown is correctly identified as the reference. Values greater than 90 are very good matches. Values less than 50 mean that substantial differences exist between the unknown and reference, and the match should be regarded with suspicion. The spectra identified in the Hazmat sample was at a very low estimated concentration, sub ppm, thus resulting in low abundances of the mass spectra ion fragments along with background noise. An operator review of the unknown and reference spectra did show a similar mass fragmentation pattern of the major peaks and was thus identified as a substituted hydrocarbon.

Table 34. VOC Raw Data of 15.8M HNO_3 Treated Samples

Sample	HS Peak 3.6 min	HS Peak 9.9 min
CC1	5,900,907	0
CC2	9,336,272	0
CC3	9,719,962	0
Ave		
HZMT1	12,040,824	104,127
HZMT2	12,693,173	73,956
HZMT3	11,300,057	65,536
Ave		
KMTC1	12,023,705	0
KMTC2	11,923,248	0
KMTC3	11,508,263	0
Ave		
PBI1	3,159,486	0
PBI2	4,243,589	0
PBI3	4,209,236	0
Ave		

Volatile Organic Compounds (VOC) Off-gas Analysis (continued)

Table 35. VOC Raw Data of Untreated Samples

Sample	HS Peak 3.6 min	HS Peak 9.9 min
Air	0	0
CCU1	0	0
PBIU1	0	0
HZMTU1	0	0
KMTCU1	0	0

Permanent Gas Analysis

Following VOC analysis above the sample vials were removed and headspace samples were manually taken with an evacuated gas container by puncturing the vial septum with a needle and opening the isolation to pull a sample into the evacuated container. The sample was transferred to a second GC and then injected into a 1 ml sample loop for permanent gas analysis. The gas chromatograph was operated using an argon carrier gas at 40psi (Ramped Pressure Mode) for 1.5 min initially then ramped at 2psi/min to 60psi final pressure. The oven was held at 40 °C for 2.5 minutes, then ramped at 15 °C/min to 200 °C. The zone temperatures were 250 °C. Data was collected using a Thermal Conductivity Detector (TCD). Data was processed using the instrument's software to determine peak areas and quantitated using a 5 point calibration curve for each component. The raw data for the samples treated with 15.8M HNO_3 are shown in Table 15 and for the untreated sample in Table 16. An error occurred with the analysis of one of the treated cheesecloth samples, therefore only two cheesecloth samples are reported.

Table 36. Raw Data from Analysis of Permanent Gases from 15.8M HNO_3 Treated Samples

Sample	He	N2	O2	CO2	CO
CC2	25.48%	54.24%	14.82%	0.09%	0.15%
CC3	25.86%	54.91%	15.01%	0.08%	0.12%
Ave	25.67%	54.58%	14.92%	0.09%	0.14%
HZMT1	25.77%	53.98%	14.66%	0.09%	0.21%
HZMT2	25.09%	54.35%	14.81%	0.10%	0.13%
HZMT3	25.49%	54.33%	14.78%	0.10%	0.13%
Ave	25.45%	54.22%	14.75%	0.10%	0.16%
KMTC1	25.23%	54.66%	14.90%	0.09%	0.16%
KMTC2	25.70%	55.11%	15.08%	0.08%	0.16%
KMTC3	25.29%	54.47%	14.85%	0.08%	0.11%
Ave	25.41%	54.75%	14.94%	0.08%	0.14%
PBI1	25.51%	54.54%	14.82%	0.11%	0.19%
PBI2	24.97%	54.53%	14.87%	0.11%	0.13%
PBI3	25.16%	54.78%	14.93%	0.11%	0.17%
Ave	25.21%	54.62%	14.87%	0.11%	0.16%

Permanent Gas Analysis (continued)

Table 37. Raw Data from Analysis of Permanent Gases from Untreated Samples

Sample	He	N2	O2	CO2	CO
Air	25.35%	54.90%	15.13%	0.08%	0.00%
CCU1	24.24%	55.14%	15.15%	0.09%	0.00%
PBIU1	24.28%	55.07%	15.19%	0.11%	0.00%
HZMTU1	25.48%	55.26%	15.21%	0.10%	0.00%
KMTCU1	25.00%	55.09%	15.10%	0.08%	0.00%

Carbon Hydrogen Nitrogen (CHN) Elemental Analysis

Samples of the untreated and HNO_3 treated cloths were analyzed for carbon, hydrogen, and nitrogen elemental composition using a Perkins Elmer 2400 Series II Elemental Analyzer configured in the CHN mode. The analyzer was configured with default temperatures of 925 °C in the combustion zone and 640 °C in the reduction zone, and using helium as the inert carrier gas. To help combust the samples, additional oxygen was added to the combustion as 1 s of flow during the OXFILL and BOOST1 stages.

The analyzer's principle of operation is to combust the sample in a tin (Sn) cup using oxygen gas (O_2) as the primary oxidizer, along with supported metal oxidants to ensure complete oxidation of the sample. The carbon, hydrogen, and nitrogen in the sample are oxidized to carbon dioxide (CO_2), water (H_2O), and nitrous oxides (NO_x), respectively. The combustion gases then pass through a copper reduction column to reduce the NO_x to molecular nitrogen (N_2) for measurement. The final gas products are collected and then chromatographically separated for measurement by thermal conductivity detection (TCD). The detector responses for CO_2, H_2O, and N_2 correspond to the elemental masses of C, H, and N comprising the sample. By calibrating detector response factors to a standard, in this work acetanilide (C_8H_9NO), the CHN elemental abundances may be determined for other samples.

The analysis of the cloths was performed using small samples (1.000 ±0.1 mg) of the cloths, either by pulling individual fibers from the sample (cheesecloth and PBI) or by using a sample punch for non-fibrous cloths (Hazmat and Kimtech). The samples were weighed into pre-tared tin sample vials, folded, and loaded onto the analyzer. Sequences were run with a series of blanks and calibration standards, along with intermittent vial blanks to check for carry-over. The raw data for the samples treated with 15.8M HNO_3 and for the untreated samples are shown in Table 17.

Table 38. Raw Data from C,H,N Analysis

Run #	ID	Weight (mg)	Vial #	Carbon (%)	Hydrogen (%)	Nitrogen (%)	ZR	Nr	Cr	Hr
Untreated										
18	CC NA1	0.924		45.29%	5.53%	0.13%	9952	9954	17346	19568
37	CC NA2	0.936		46.21%	6.57%	0.17%	10270	10268	17898	20351
19	HZMT NA1	1.069		85.60%	17.59%	0.21%	9952	9959	26131	32849
38	HZMT NA2	1.020		81.03%	18.60%	0.55%	10276	10294	24891	31552
20	KMTC NA1	1.125		82.46%	18.41%	0.12%	9966	9969	26364	33711
39	KMTC NA2	1.066		78.13%	19.45%	0.49%	10266	10282	24990	32231
21	PBI NA1	0.970		10.48%	2.39%	8.51%	9968	10378	12167	13473
40	PBI NA2	0.895		11.08%	3.25%	8.22%	10275	10634	12369	13758
Treated										
23	CC A1	1.042		47.87%	3.73%	1.68%	9946	10030	18841	20662
44	CC A2	1.034		41.68%	2.93%	0.95%	10260	10299	17901	19328
24	HZMT A1	1.132		78.07%	16.60%	0.67%	9951	9985	25604	32320
45	HZMT A2	0.902		76.95%	17.16%	0.51%	10248	10261	22516	28031
25	KMTC A1	1.025		82.35%	17.18%	0.51%	9963	9985	24902	31229
46	KMTC A2	0.959		63.95%	17.80%	1.56%	10262	10327	21153	27191
26	PBI A1	0.991		13.51%	1.49%	11.49%	9968	10535	12894	13923
47	PBI A2	1.006		13.75%	2.49%	12.25%	10279	10887	13314	14571

Table 3.8 Raw Data from C,H,N Analysis (continued)

Run #	ID	Weight (mg)	Vial #	Carbon (%)	Hydrogen (%)	Nitrogen (%)	ZR	Nr	Cr	Hr
STD										
17	STD1	1.018		76.32%	6.86%	13.45%	9935	10618	24348	27187
27	STD3	1.086		64.07%	5.34%	10.01%	9955	10496	22791	25242
36	STD4	0.843		70.50%	6.79%	9.46%	10267	10657	21148	23462
51	STD5	1.007		75.15%	4.69%	10.10%	10261	10761	24124	26109
52	STD6	0.896		72.19%	5.62%	9.70%	10263	10689	22108	24196
Blank										
41	VIAL 1	1.000		1.95%	-0.15%	0.24%	10271	10273	10599	10984
42	VIAL 2	1.000		1.61%	-0.48%	0.10%	10267	10262	10529	10807
43	VIAL 3	1.000		1.32%	-0.60%	0.08%	10264	10258	10473	10710
48	VIAL 4	1.000		1.46%	-0.28%	0.30%	10274	10279	10519	10862
49	VIAL 5	1.000		1.36%	-0.60%	0.14%	10267	10264	10486	10724
50	VIAL 6	1.000		2.14%	-0.71%	0.12%	10263	10259	10620	10821
53	VIAL 7	1.000		1.45%	-0.35%	0.26%	10264	10267	10506	10825

Fourier-Transform Infrared Spectroscopy (FT-IR) Attenuated Total Reflection (ATR)

Samples treated with concentrated nitric acid as well as untreated samples were analyzed using a Thermo Scientific Nicolet iS50 FTIR with the built in Touchpoint (single bounce) ATR. The ATR attachment used a diamond crystal with a ZnSe lens. The IR beam angle was 42°. Prior to sample analysis, a background was taken with the press arm up and a clean diamond plate area. Then the sample was placed on the stage in contact with the crystal surface and it was scanned to obtain a spectrum. Results for the treated and untreated samples are shown in the main body of this report.

Differential Scanning Calorimetry (DSC) Analysis

Samples of the untreated and HNO_3 treated cloths were analyzed for mass loss and enthalpy changes using a Netzsch Model 410 Analyzer configured in the DSC mode with MS analysis for gas evolution. The carrier gas was air to simulate combustion reactions. Data was collected using the instruments software and analyzed with a Proteus software to determine mass loss and enthalpy values. The two profiles used are described in the body of the report. Analyses not shown in the body of the report are presented below.

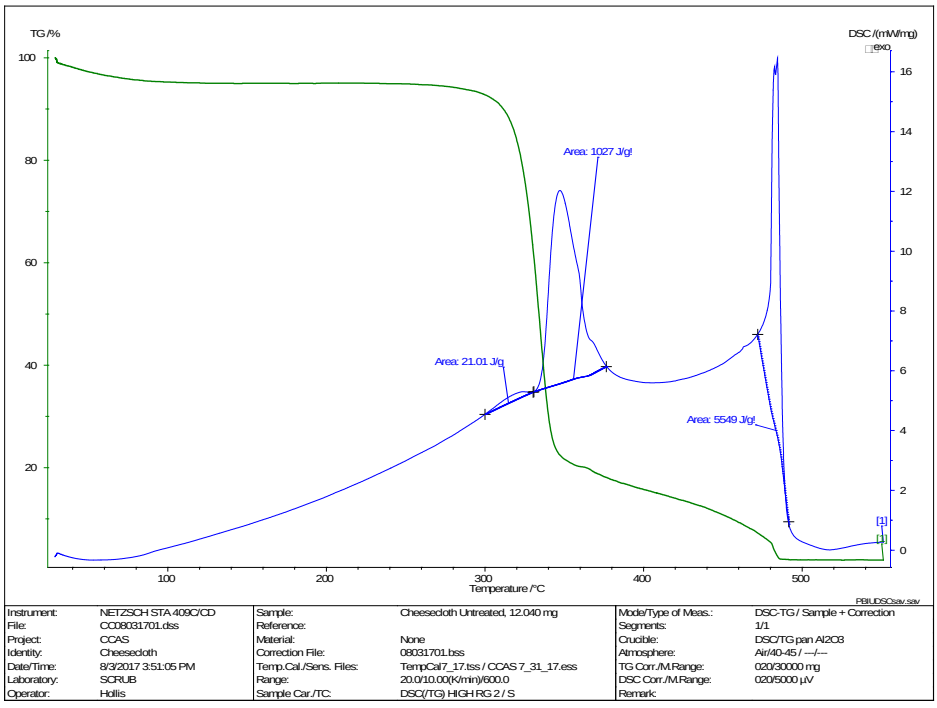
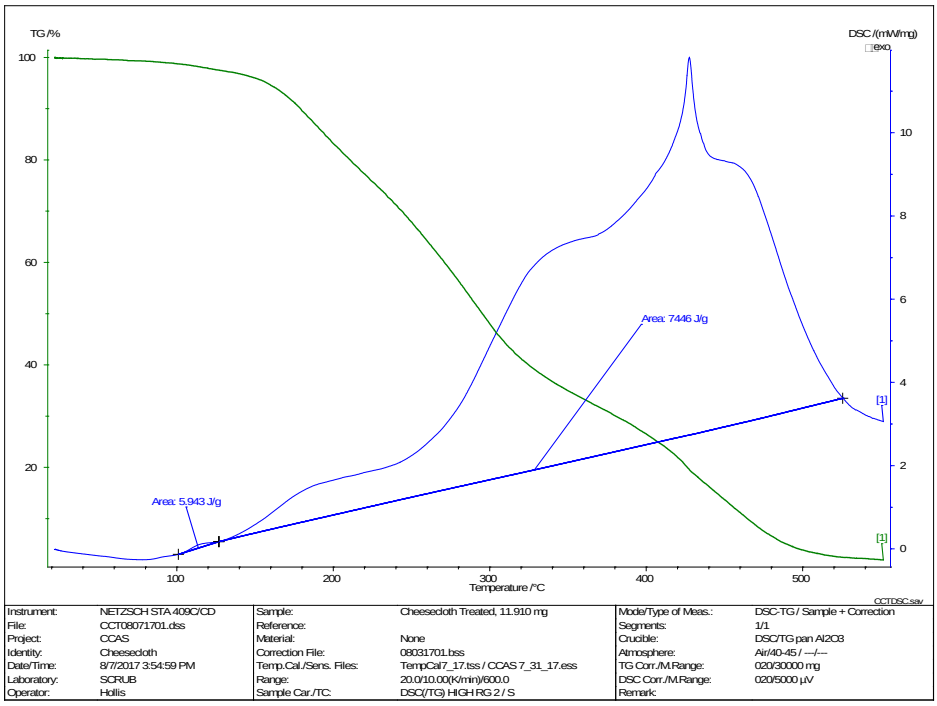
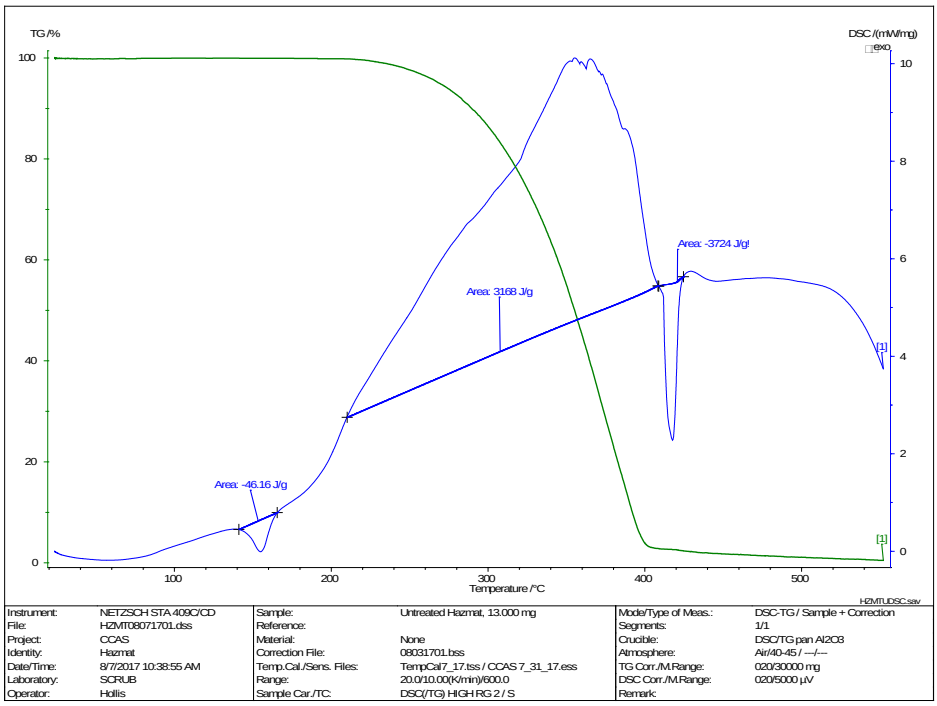


Figure 61. DSC of Untreated Cheesecloth (profile 2)



Instrument:	NETZSCH STA 409C/CD	Sample:	Cheesecloth Treated, 11.910 mg	Mode/Type of Meas.:	DSC-TG / Sample + Correction
File:	CCT08071701.dss	Reference:		Segments:	1/1
Project:	CCAS	Material:	None	Crucible:	DSC/TG pan Al2O3
Identity:	Cheesecloth	Correction File:	08031701.bss	Atmosphere:	Air/40-45 / ---/---
Date/Time:	8/7/2017 3:54:59 PM	Temp.Cal./Sens. Files:	TempCal7_17.tss / CCAS 7_31_17.ess	TG Corr./M.Range:	020/30000 mg
Laboratory:	SCRUB	Range:	20.0/10.00(K/min)/600.0	DSC Corr./M.Range:	020/5000 µV
Operator:	Hollis	Sample Car./TC:	DSC(/TG) HIGH RG 2 / S	Remark:	

Figure 62. DSC of Treated Cheesecloth (profile 2)



Instrument:	NETZSCH STA 409C/CD	Sample:	Untreated Hazmat, 13.000 mg	Mode/Type of Meas.:	DSC-TG / Sample + Correction
File:	HZMT08071701.dss	Reference:		Segments:	1/1
Project:	CCAS	Material:	None	Crucible:	DSC/TG pan Al2O3
Identity:	Hazmat	Correction File:	08031701.bss	Atmosphere:	Air/40-45 / ---/---
Date/Time:	8/7/2017 10:38:55 AM	Temp.Cal./Sens. Files:	TempCal7_17.tss / CCAS 7_31_17.ess	TG Corr./M.Range:	020/30000 mg
Laboratory:	SCRUB	Range:	20.0/10.00(K/min)/600.0	DSC Corr./M.Range:	020/5000 µV
Operator:	Hollis	Sample Car./TC:	DSC(/TG) HIGH RG 2 / S	Remark:	

Figure 63. DSC of Untreated Hazmat (profile 2)

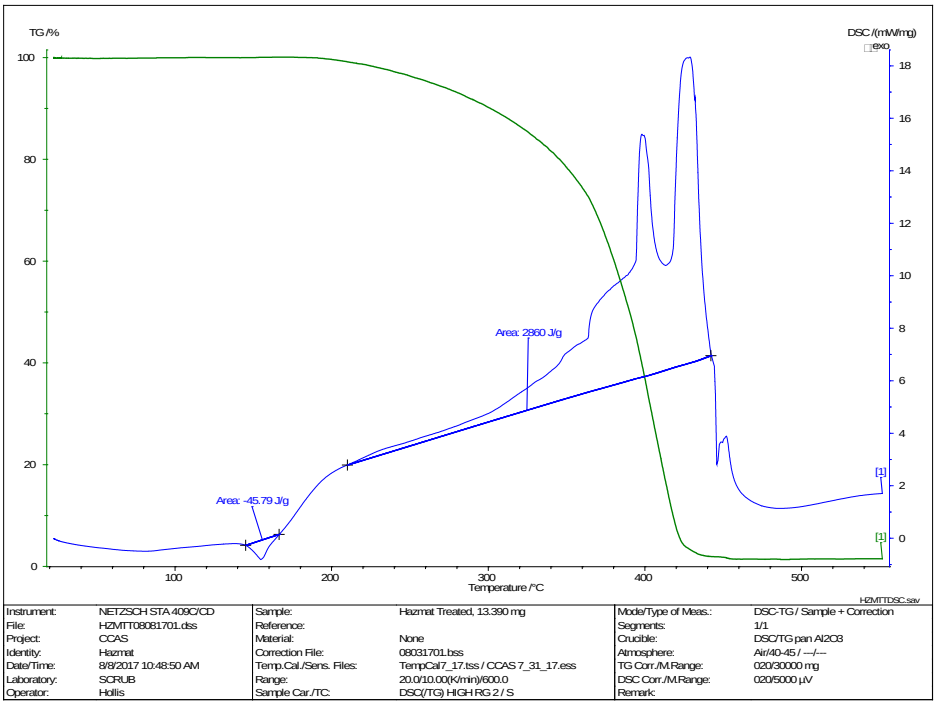


Figure 64. DSC of Treated Hazmat (profile 2)

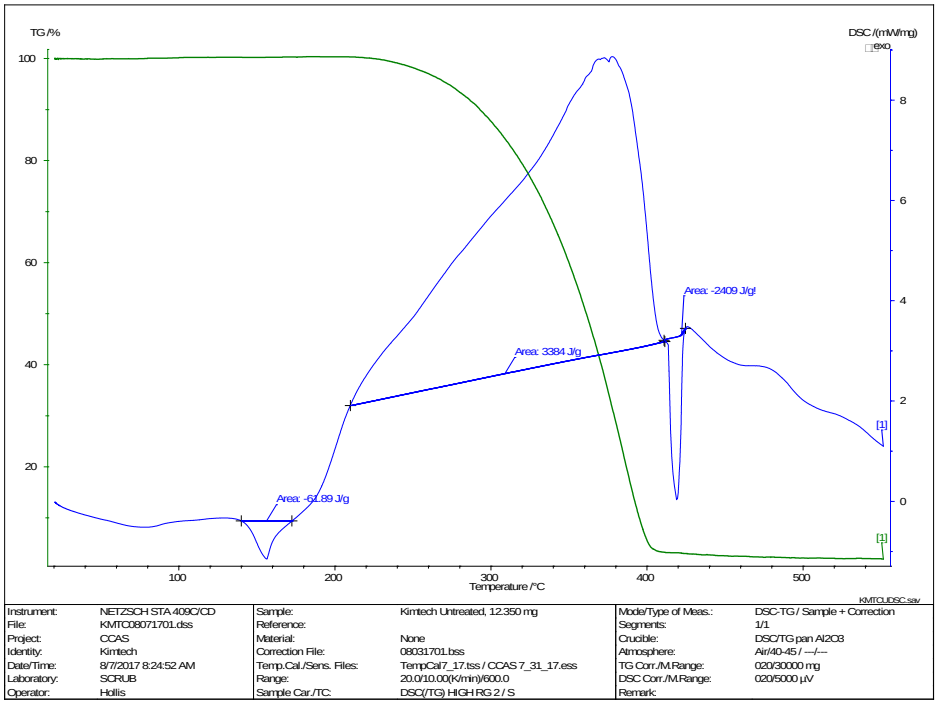


Figure 65. DSC of Untreated Kimtech (profile 2)

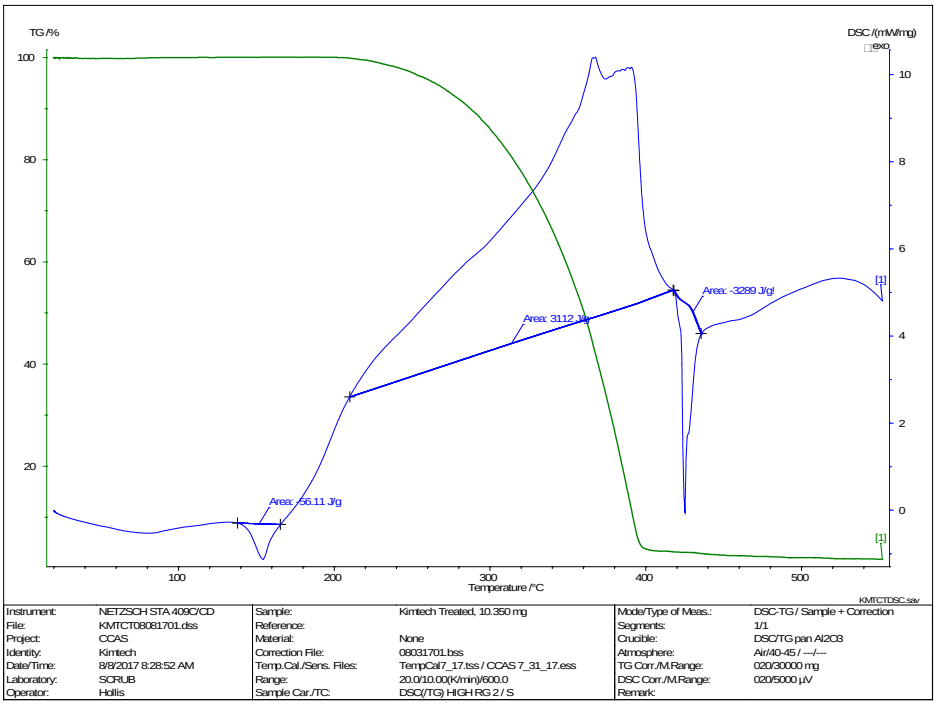


Figure 66. DSC of Treated Kimtech (profile 2)

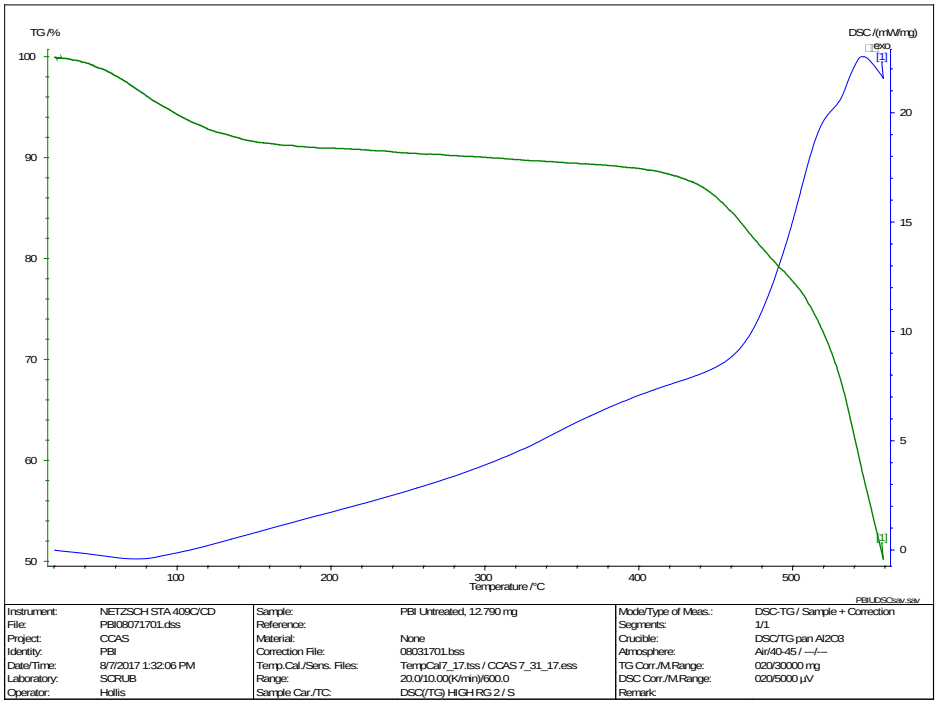
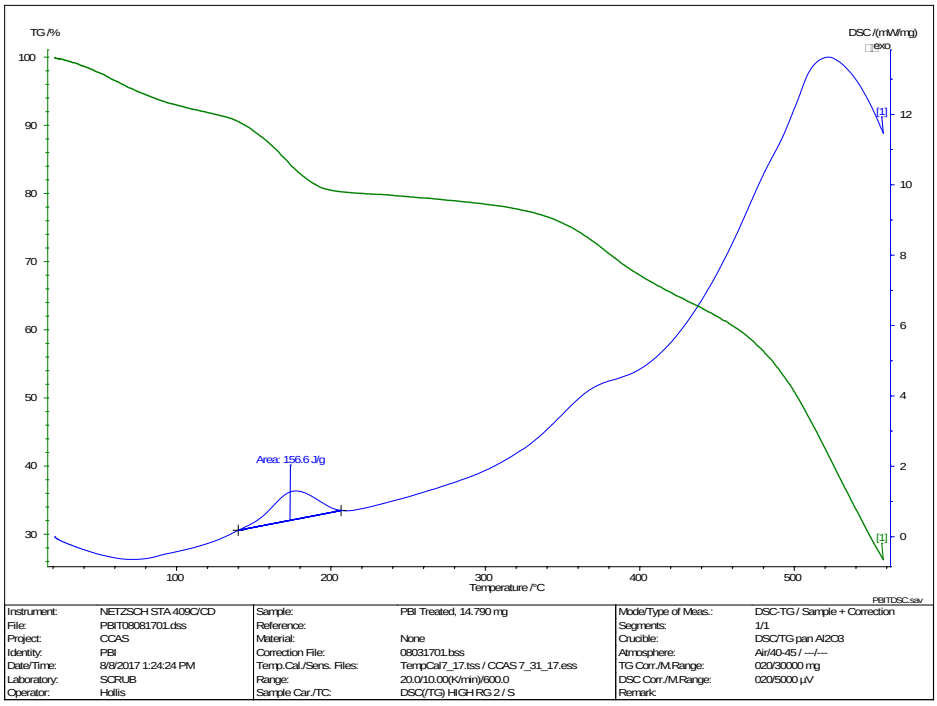


Figure 67. DSC of Untreated PBI (profile 2)



Instrument:	NETZSCH STA 409C/CD	Sample:	PBI Treated, 14.790 mg	Mode/Type of Meas.:	DSC-TG / Sample + Correction
File:	PBIT08081701.dss	Reference:		Segments:	1/1
Project:	CCAS	Material:	None	Crucible:	DSC/TG pan Al2O3
Identity:	PBI	Correction File:	08031701.bss	Atmosphere:	Air/40-45 / ---/---
Date/Time:	8/8/2017 1:24:24 PM	Temp.Cal./Sens. Files:	TempCal7_17.tss / CCAS 7_31_17.ess	TG Corr./M.Range:	020/30000 mg
Laboratory:	SCRUB	Range:	20.0/10.00(K/min)/600.0	DSC Corr./M.Range:	020/5000 µV
Operator:	Hollis	Sample Car./TC:	DSC(/TG) HIGH RG 2 / S	Remark:	

Figure 68. DSC of Treated PBI (profile 2)

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